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L29 ANSWER 1 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2004:348011 HCAPLUS Full-text

DOCUMENT NUMBER:

140:356948

TITLE:

Catalytic addition reaction for the production

of 3-(methylthio)propanal from mercaptomethane

and acrolein

INVENTOR(S):

Rey, Patrick

PATENT ASSIGNEE(S):

Adisseo France S.A.S., Fr.

SOURCE:

Eur. Pat. Appl., 10 pp. CODEN: EPXXDW

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

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OTHER SOURCE(S): CASREACT 140:356948

AB A process for the production of 3-(methylthio)propanal comprises reacting mercaptomethane and acrolein in the presence of a catalyst comprising an organic base such as an N-alkylmorpholine (e.g., 4-methylmorpholine).

IT 64-19-7, Acetic acid, uses

(addition reaction catalysts in the production of 3-(methylthio)propanal from mercaptomethane and acrolein)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

H3C-SH

RN 107-02-8 HCAPLUS CN 2-Propenal (9CI) (CA INDEX NAME)

H2C---CH--CH---O

 $MeS-CH_2-CH_2-CHO$

IC ICM C07C319-18
ICS C07C323-22

CC 23-14 (Aliphatic Compounds)
Section cross-reference(s): 45, 67

IT 64-18-6, Formic acid, uses 64-19-7, Acetic acid, uses 79-09-4, Propanoic acid, uses 100-74-3, 4-Ethylmorpholine 107-92-6, Butyric acid, uses 109-02-4, 1-Methylmorpholine (addition reaction catalysts in the production of 3-(methylthio)propanal from mercaptomethane and acrolein)

TT 74-93-1, Mercaptomethane, reactions 107-02-8,
Acrolein, reactions

(catalytic addition reaction for the production of

3-(methylthio)propanal from mercaptomethane and acrolein) IT 3268-49-3P, 3-(Methylthio)propanal

(catalytic addition reaction for the production of

3-(methylthio)propanal from mercaptomethane and acrolein)
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE

FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 2 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2004:269858 HCAPLUS Full-text DOCUMENT NUMBER: 140:287102

TITLE: Method for producing 3-methylthiopropanal from

acrolein and methyl mercaptan

INVENTOR(S):

Shiozaki, Tetsuya; Haga, Toru

PATENT ASSIGNEE(S):

Sumitomo Chemical Company, Limited, Japan

SOURCE:

U.S. Pat. Appl. Publ., 4 pp.

CODEN: USXXCO.

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	PA:	rent :	NO.			KINI	D -	DATE		AP	PL	ICAT:	ION :	NO.		DATE
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OTHER SOURCE(S): CASREACT 140:287102

AB 3-Methylthiopropanal is produced in high yield and selectivity by supplying acrolein and Me mercaptan together or sequentially with an acidic compound (e.g., acetic acid) and a basic compound (e.g., pyridine) into a reaction system to react the acrolein with the Me mercaptan, where the basic compound is used in an amount of about 0.3 mol or less per mol of the acidic compound 74-93-1, Methyl mercaptan, reactions 107-02-8,

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0927

Acrolein, reactions

(method for producing 3-methylthiopropanal from acrolein and Me mercaptan)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

RN 107-02-8 HCAPLUS

CN 2-Propenal (9CI) (CA INDEX NAME)

IT 3268-49-3P, 3-Methylthiopropanal

(method for producing 3-methylthiopropanal from acrolein and Me mercaptan)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IT 64-19-7, Acetic acid, uses

(method for producing 3-methylthiopropanal from acrolein and Me mercaptan using)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

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IC ICM C07C323-22

INCL 514041000

CC 23-14 (Aliphatic Compounds)

Section cross-reference(s): 45.

IT 74-93-1, Methyl mercaptan, reactions 107-02-8,

Acrolein, reactions

(method for producing 3-methylthiopropanal from acrolein and Me mercaptan)

IT 3268-49-3P, 3-Methylthiopropanal

(method for producing 3-methylthiopropanal from acrolein and Me mercaptan)

IT **64-19-7**, Acetic acid, uses 110-86-1, Pyridine, uses (method for producing 3-methylthiopropanal from acrolein and Me mercaptan using)

L29 ANSWER 3 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2003:639035 HCAPLUS Full-text

DOCUMENT NUMBER:

139:166201

TITLE:

Process for purification of acrolein

INVENTOR(S):

Gros, Georges; Garrait, Michel; Rey, Patrick

PATENT ASSIGNEE(S):

Aventis Animal Nutrition S.A., Fr.

SOURCE:

Fr. Demande, 20 pp.

DOCUMENT TYPE:

CODEN: FRXXBL

LANGUAGE:

Patent

FAMILY ACC. NUM. COUNT:

French

PARTIE ACC. NOM. COL

PATENT INFORMATION:

PATENT NO.

KIND DATE

APPLICATION NO.

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FR 2835831	A1 20030815	FR 2002-1686	
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The present invention thus has as an aim a continuous process of purification of the acrolein in which: (1) an aqueous acrolein solution deprived of noncondensable gas is fed into a distillation column; (2) an aqueous mixture is drawn off from the bottom; (3) a mixture based on acrolein and water is drawn off from the head; (4) the head fraction is cooled to sep. the water from an acrolein-rich gas, and (5) acrolein is isolated from the gas from (4). The resulting acrolein is suitable for manufacture of 3-methylthiopropionaldehyde by reaction with Me mercaptan.

IT 3268-49-3P, 3-Methylthiopropionaldehyde

(purification of acrolein by distillation of aqueous compns. for manufacture of

methylthiopropionaldehyde by reaction with Me mercaptan)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME).

MeS-CH2-CH2-CHO

IT 74-93-1, Methyl mercaptan, reactions

(purification of acrolein by distillation of aqueous compns. for manufacture of

methylthiopropionaldehyde by reaction with Me mercaptan)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IC ICM C07C045-82

ICS C07C047-22; C07C319-28; C07C321-18; A23K001-22

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes) Section cross-reference(s): 23

IT 3268-49-3P, 3-Methylthiopropionaldehyde

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(purification of acrolein by distillation of aqueous compns. for manufacture of

methylthiopropionaldehyde by reaction with Me mercaptan)

IT 74-93-1, Methyl mercaptan, reactions

(purification of acrolein by distillation of aqueous compns. for manufacture of

methylthiopropionaldehyde by reaction with Me mercaptan)

REFERENCE COUNT:

THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 4 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2001:112320 HCAPLUS Full-text DOCUMENT NUMBER: 134:164826 TITLE: Manufacture of acrolein and acrolein derivatives from Diels-Alder reaction or Michael addition Etzkorn, William George; Galley, Richard A.; INVENTOR(S): Snead, Thomas E.; Brockwell, Jonathan Lester; Young, Mark Anderson; Maher, John Michael; Warren, Barbara Knight PATENT ASSIGNEE(S): Union Carbide Chemicals and Plastics Technology Corporation, USA U.S., 11 pp., Cont.-in-part of WO9736848. SOURCE: CODEN: USXXAM DOCUMENT TYPE: Patent LANGUAGE: English FAMILY ACC. NUM. COUNT: 3 PATENT INFORMATION: PATENT NO. KIND DATE APPLICATION NO. DATE ---------US 6187963 B1 20010213 US 1998-169798 1998 1009 <--WO 9736848 19971009 WO 1997-US5100 A1 1997 0327 <--W: GH, KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG RW: AU, BB, BG, BR, CA, CN, CZ, HU, IS, JP, KP, KR, LK, LR, LV, MK, MX, NO, NZ, PL, SG, SI, TR, TT, US, UZ, VN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM Al 19990120 EP 1997-917687 1997 0327 <--20030521 EP 891316 B1 R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI PRIORITY APPLN. INFO.: EP 1997-917687 1997 0327 <--WO 1997-US5100 1997 0327 <---US 1996-14507P 1996 0401 <--US 1996-14510P 1996 0401

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US 1996-14678P

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AB A process for producing an acrolein derivative comprises (i) passing a propylene feed stream comprising propylene, oxygen, and a recycle gas comprising propane, oxygen, and at least one of carbon monoxide and carbon dioxide to an acrolein reaction zone wherein the propylene feed stream is contacted with an acrolein reaction catalyst at conditions effective to promote the formation of acrolein to provide an acrolein effluent stream comprising acrolein, propane, acetaldehyde and water; (ii) passing the acrolein effluent stream to an acrolein separation zone wherein the acrolein effluent stream is partially condensed to provide a liquid acrolein product stream comprising acrolein, acetaldehyde, and water and a recycle gas stream comprising the recycle gas; (iii) passing the acrolein product stream and a co-reactant capable of undergoing a Diels-Alder reaction or Michael addition with acrolein to an acrolein derivative reaction zone and contacting the acrolein and co-reactant under conditions effective to convert the acrolein and the co-reactant into an acrolein derivative; and (iv) recycling at least a portion of the recycle gas stream to the acrolein reaction zone. The process is characterized in that the propylene feed stream comprises an amount of propane of from about 5 to 70 volume% and effective to provide a propylene-toacrolein reaction efficiency of from about 75 to 90 mol%.

IT 3268-49-3P, 3-(Methylthio)propanal

(manufacture of acrolein and acrolein derivs. from Diels-Alder reaction or Michael addition)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

RN 107-02-8 HCAPLUS

CN 2-Propenal (9CI) (CA INDEX NAME)

 $H_2C \longrightarrow CH - CH \longrightarrow O$

IT 64-19-7, Acetic acid, reactions 74-93-1, Methyl
 mercaptan, reactions
 (manufacture of acrolein and acrolein derivs. from Diels-Alder
 reaction or Michael addition)
RN 64-19-7 HCAPLUS
CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

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c07c027-10; c07c045-27; c07c045-32 INCL 568469900 CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes) Section cross-reference(s): 23 IT 100-73-2P, 2-Formyl-3,4-dihydro-2H-pyran 108-99-6P, 110-86-1P, Pyridine, preparation B-Picoline 111-30-8P, 504-63-2P, 1,3-Propanediol Glutaraldehyde 1321-16-0P, Tetrahydrobenzaldehyde 3268-49-3P; 3-(Methylthio) propanal 31906-04-4P, 4-(4-Hydroxy-4-methylpentyl)-3cyclohexene-1-carboxaldehyde 75454-86-3P 84315-07-1P (manufacture of acrolein and acrolein derivs. from Diels-Alder reaction or Michael addition) ΙT 107-02-8P, Acrolein, preparation 2134-29-4P, 3-Hydroxypropionaldehyde 4454-05-1P, 2-Methoxy-3,4-dihydro-2Hpyran (manufacture of acrolein and acrolein derivs. from Diels-Alder reaction or Michael addition) 56-81-5, 1,2,3-Propanetriol, reactions 57-55-6, Propylene IT glycol, reactions 64-17-5, Ethanol, reactions 64-19-7, 65-85-0, Benzoic acid, reactions Acetic acid, reactions 67-56-1, Methanol, reactions 67-63-0, Isopropanol, reactions 74-93-1, Methyl mercaptan, reactions 79-09-4, Propionic acid, reactions 106-99-0, Butadiene, reactions 107-18-6, Allyl 107-21-1, Ethylene glycol, reactions alcohol, reactions 107-25-5, Methyl vinyl ether 108-24-7, Acetic anhydride 115-07-1, Propylene, reactions 115-77-5, Pentaerythritol, 123-35-3, Myrcene 543-39-5 reactions 7664-41-7, Ammonia, 7732-18-5, Water, reactions reactions 30700-92-6 (manufacture of acrolein and acrolein derivs. from Diels-Alder reaction or Michael addition) REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT L29 ANSWER 5 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 2000:909250 HCAPLUS Full-text DOCUMENT NUMBER: 134:43711 TITLE:

Oxidative processes for the manufacture of

acrolein from propylene and oxygen

INVENTOR(S):

Etzkorn, William George; Brockwell, Jonathan Lester; Young, Mark Anderson; Maher, John

Michael; Warren, Barbara Knight

PATENT ASSIGNEE(S):

Union Carbide Chemicals and Plastics

Technology Corporation, USA

SOURCE:

U.S., 10 pp., Cont.-in-part of Appl. No.

PCT/US97/05100. CODEN: USXXAM

DOCUMENT TYPE:

Patent English

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

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AΒ Acrolein is produced in high yield and selectivity in a process comprising: (i) passing a propylene feedstream comprising propylene, oxygen and a recycle gas comprising propane, oxygen and carbon monoxide and/or carbon dioxide to an acrolein reaction zone where the propylene feedstream is contacted with an acrolein reaction catalyst to provide an acrolein effluent stream comprising acrolein, propane, acetaldehyde and water; (ii) passing the acrolein effluent stream to an acrolein separation zone where the acrolein effluent stream is partially condensed to provide a liquid acrolein product stream comprising acrolein, acetaldehyde and water and a recycle gas stream comprising the recycle gas; and (iii) recycling a portion of the recycle gas stream to the acrolein reaction zone. The propylene feedstream comprises 5-70 volume% propane and is effective to provide a propylene-to-acrolein reaction efficiency of 75-90 mol%. The presence of propane in the propylene-toacrolein reaction can enhance the efficiency of the processes. IT

74-93-1, Methanethiol, reactions

(addition reactions of acrolein with)

RN74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

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IT
     3268-49-3P
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(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

C07C045-32

INCL 568469900

45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes) Section cross-reference(s): 23, 48

74-93-1, Methanethiol, reactions 106-99-0, Butadiene, reactions 543-39-5

(addition reactions of acrolein with)

IT 78-19-3P 100-73-2P, 2-Formyl-3,4-dihydro-2H-pyran 504-63-2P, 1,3-Propanediol 1321-16-0P, Tetrahydrobenzaldehyde 2806-84-0P, 3-(Methoxy)propionaldehyde 3268-49-3P 4454-05-1P, 2-Methoxy-3,4-dihydro-2H-pyran 31906-04-4P, 4-(4-Hydroxy-4methylpentyl)-3-cyclohexene-1-carboxaldehyde 84315-07-1P (preparation of)

REFERENCE COUNT:

13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 6 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

2000:289080 HCAPLUS Full-text

DOCUMENT NUMBER:

132:309995

TITLE:

Processes for the manufacture of

3-(methylthio)propanal

INVENTOR(S):

Brockwell, Jonathan L.; Young, Mark A.; Etzkorn, William G.; Warren, Barbara K.;

Maher, John M.

PATENT ASSIGNEE(S):

Union Carbide Chemicals & Plastics Technology

Corporation, USA

SOURCE:

U.S., 12 pp. CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE 	APPLICATION NO.	DATE
us 6057481	A	20000502	US 1998-155750	1998
WO 9736848	Al	19971009	`< WO 1997-US5100	1001 1997 0327

<--W: GH, KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG,

CI, CM, GA, GN, ML, MR, NE, SN, TD, TG

RW: AU, BB, BG, BR, CA, CN, CZ, HU, IS, JP, KP, KR, LK, LR,

AU		BY,	KG,	ΚZ,	NZ, P MD, R 19	U, TJ	J,	TM		тт, .997-2			VN,	AM,
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														0327
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AT	240924			E	20	03061	.5	ΓA	1	.997-9	9176	37		
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PRIORITY	APPLN.	INFO	. :					US	3 1	.996-:	1450	7 P	1	=
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								WC	1	.997–เ	JS510	00	V	V
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AB A process for the conversion of propylene to 3- (methylthio)propanal (I) by converting propylene to acrolein and converting the acrolein with Me mercaptan to I is described. The processes utilize oxygen and recycle propane to the acrolein reactor. The process feeds can comprise, propane, propylene or their mixts. The presence of propane in the propylene-to-acrolein reaction can enhance the efficiency of the processes.

IT 3268-49-3P, 3-(Methylthio)propanal

(processes for the manufacture of 3-(methylthio)propanal)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

H3C-SH

INCL 568041000

45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes) Section cross-reference(s): 23, 48

IT**3268-49-3P**, 3-(Methylthio)propanal

(processes for the manufacture of 3-(methylthio)propanal)

74-93-1, Methyl mercaptan, reactions 74-98-6, Propane, IT reactions

(processes for the manufacture of 3-(methylthio)propanal)

REFERENCE COUNT:

23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L29 ANSWER 7 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1999:450926 HCAPLUS Full-text

DOCUMENT NUMBER:

131:89346

TITLE:

Continuous process for the preparation of 3-(methylthio)propanal from acrolein and

methyl mercaptan

INVENTOR(S):

Hsu, Yung C.; Ruest, Dennis A. Novus International, Inc., USA

SOURCE:

U.S., 26 pp. CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT ASSIGNEE(S):

PATENT NO.	KIND	.DATE 	APPLICATION NO.	DATE
 US 5925794	A .	19990720	US 1996-668572	1996 0620
US 5352837	А	19941004	< US 1993-73763	1993
US 5637766	А	19970610	< US 1995-557699	1995
CN 1188470	Α	19980722	< CN 1996-194943	1113 1996
CN 1120834 US 6031138	B A	20030910 20000229	< US 1998-102025	0621
03 0031130		20000223	<	1998 0622
บร 6320076	В1	20011120	US 1999-470407	1999 1222
PRIORITY APPLN. INFO.:			< US 1993-73763	A2 1993 0608
			< US 1994-273216	B1

			1994 0711
< US 199	5-421P	P	1995 0622
	5-557699	A2	1995 1113
< US 199	6-667099	В1	1996 0620
< US 199	6-668572	В1	1996 0620
< US 199	8-102025	Ä3	1998 0622

AB 3-(Methylthio)propanal (I) is prepared in a continuous process in which a liquid reaction medium (containing I, Me mercaptan, and an addition reaction catalyst) is contacted with a gaseous acrolein feed stream (containing acrolein vapor and noncondensable gas) in a gas-liquid contact zone. Acrolein is transferred from the acrolein feed stream to the reaction medium and reacted with Me mercaptan in that medium to produce a liquid reaction product containing I. The noncondensable gas is separated from the liquid reaction product, the reaction product is divided into a product fraction and a circulating fraction, and the circulating fraction is recycled to the gas-liquid contact zone. Process flow diagrams are presented.

IT 3268-49-3P, 3-(Methylthio)propanal

(continuous process for the preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

TT 74-93-1, Methyl mercaptan, reactions
 (continuous process for the preparation of 3-(methylthio)propanal
 from acrolein and Me mercaptan)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

 ${\tt H3C-SH}$

IC ICM C07C319-00
INCL 568041000
CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)

Section cross-reference(s): 23, 48

IT 107-02-8P, 2-Propenal, preparation 3268-49-3P,

3-(Methylthio)propanal 59121-24-3P, 4-(Methylthio)butyronitrile (continuous process for the preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

74-90-8, Hydrogen cyanide, reactions **74-93-1**, Methyl ΙT mercaptan, reactions 115-07-1, 1-Propene, reactions

(continuous process for the preparation of 3-(methylthio)propanal

from acrolein and Me mercaptan)

40

REFERENCE COUNT:

THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 8 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 1998:523142 HCAPLUS Full-text

DOCUMENT NUMBER:

CORPORATE SOURCE:

129:275013

TITLE:

Quantification of potent odorants in Camembert cheese and calculation of their odor activity

values

AUTHOR(S):

Kubickova, J.; Grosch, W. Deutsche Forschungsanstalt fur

SOURCE:

Lebensmittelchemie, Garching, D-85748, Germany

International Dairy Journal (1998),

8(1), 17-23

CODEN: IDAJE6; ISSN: 0958-6946

PUBLISHER:

Elsevier Science Ltd. Journal

DOCUMENT TYPE: LANGUAGE:

English

Sixteen neutral compds., which had been screened in preceding dilution expts. as potent odorants of Camembert cheese, were quantified by stable isotope dilution assays in two samples of this cheese. In addition, seven volatile acids were determined by using conventional methods. The odor activity values (OAVs) of the compds. were calculated by dividing their concns. in the cheese samples by their odor thresholds in sunflower oil (neutral compds.) and water (acids). In the class of the neutral odorants, the highest OAVs were found for methanethiol, methional and di-Me sulfide all of which contributed to the sulfury, garlic-like note in the odor profile of Camembert. Although the OAV of 1-octen-3-ol was relatively low, this alc. and the corresponding ketone were responsible for the mushroom-like note. In the acidic fraction, acetic, butyric and capric acid showed the highest OAVs.

IT 64-19-7, Acetic acid, biological studies 74-93-1

, Methanethiol, biological studies 3268-49-3, Methional (quantification of potent odorants in Camembert cheese and calcn. of odor activity values)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

RN 74-93-1 HCAPLUS

Methanethiol (8CI, 9CI) (CA INDEX NAME)

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RN
    3268-49-3 HCAPLUS
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CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

CC 17-8 (Food and Feed Chemistry) 60-12-8, 2-Phenylethanol 64-19-7, Acetic acid, IT

66-25-1, Hexanal 74-93-1, biological studies 75-07-0, Acetaldehyde, Methanethiol, biological studies biological studies 75-18-3, Dimethyl sulfide 79-09-4, Propionic acid, biological studies 103-45-7, Phenethyl acetate 107-92-6, Butyric acid, biological studies 112-12-9, 124-07-2, Caprylic acid, biological studies 2-Undecanone 142-62-1, Caproic acid, biological studies 334-48-5, Decanoic 503-74-2, 3-Methylbutyric acid 431-03-8, 2,3-Butanedione 705-86-2, δ -Decalactone 590-86-3, 3-Methylbutanal 1618-26-4, Methylene bis(methylsulfide) 3268-49-3,

Methional 3391-86-4, 1-Octen-3-ol 3658-80-8, Dimethyl trisulfide 4312-99-6, 1-Octen-3-one 85213-22-5,

2-Acetyl-1-pyrroline

(quantification of potent odorants in Camembert cheese and calcn. of odor activity values)

REFERENCE COUNT:

28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L29 ANSWER 9 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1998:31172 HCAPLUS Full-text

DOCUMENT NUMBER:

128:114715

TITLE:

Processes for the preparation of

3-(methylthio)propanal and

2-hydroxy-4-(methylthio)butanenitrile Blackburn, Thomas F.; Pellegrin, Paul F.

INVENTOR(S): PATENT ASSIGNEE(S):

Novus International, Inc., USA

U.S., 9 pp., Cont.-in-part of U.S. 5,663,409. SOURCE:

CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

KIND	DATE .	APPLICATION NO.	DATE
Α	19980106	US 1995-581249	1005
			1995 1229
		<	
Α	19970902	US 1995-476356	
		•	1995
			0607 .
		<	
Α	19960820	ZA 1996-4335	
	A A	A 19970902	A 19980106 US 1995-581249 < A 19970902 US 1995-476356 <

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	WO	9640	531			A1		1996	1219		WO	1996-	-US90	60			1006
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		W:	AL.	AM.	AT.	AU.	AZ.	BB.	BG.	BR.	ВУ	, CA,	CH.	CN.	CZ.	DE	Ξ.
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		RW:	KE,	LS,	MW,	SD,	SZ,	UG,	AT,	BE,	CF	H, DE,	DK,	ES,	FI,	FF	٦,
							LU,	MC,	NL,	PT,	SE	E, BF,	ВJ,	CF,	CG,	C1	Ι,
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	AU	96598	373			A1		1996	1230		ΑU	1996-	-5987	3			1000
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		8303		•		A1					ΕP	1996-	-9172	22			
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	ΕP	8303						2001				•					
				DE,	DK,							, MC,					
	CN	1189	318			Α		1998	3805	1	CN	1996-	1951	90			1006
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	CN	1092	184			В		2002	1009			`					
		1151				Т2		1999			JΡ	1997-	5014	71			
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	RU	2173	581			C2		2001	0920		RU	1998-	-1002	20			100'6
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	ES	2160	319			Т3		2001	1116		ES	1996-	9172	22			
		2100	,,,					2001				1330	J 1 , 2 ,				1996
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	PΤ	8303	11			T		2001	1228		PT	1996-	9172	22			
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	CM	15100	120			А	•	2004	7777		CN	< 2002-	2002	12641	. 7		
	CIV	1310	330			A		2004	,,,,		CIA	2002-	2002	1204.	<i>3 1</i>		1996
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												<					1229
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OTHER SOURCE(S): CASREACT 128:114715; MARPAT 128:114715

AB A catalytic processes for the preparation of 3-(methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile using novel addition catalysts is described. The novel addition catalysts include: triisopropanolamine, nicotinamide, imidazole, benzimidazole, 2-fluoropyridine, poly-4-vinylpyridine, 4-dimethylaminopyridine, picoline, pyrazine, trialkylamines, and tertiary amines. E.g., reaction of MeSH and acrolein in presence of poly-4-vinylpyridine gave 89.0% 3-(methylthio)propanal. The aldehyde product, containing the poly-4-vinylpyridine catalyst, was converted to the nitrile in the same reactor by treatment with HCN. The yield of nitrile was 72.9%.

IT 3268-49-3P, 3-(Methylthio)propanal

(preparation of (methylthio)propanal and hydroxy(methylthio)butanenitrile)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

H3C-SH

IC ICM C07C323-22

ICS C07C253-00; C07C253-30; C07C319-20

INCL 558351000

CC 23-9 (Aliphatic Compounds)

Section cross-reference(s): 45

'IT 3268-49-3P, 3-(Methylthio)propanal

(preparation of (methylthio)propanal and

hydroxy(methylthio)butanenitrile)

TT 74-93-1, Methyl mercaptan, reactions 107-02-8, Acrolein, reactions

(preparation of (methylthio) propanal and

hydroxy(methylthio)butanenitrile)

REFERENCE COUNT:

37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L29 ANSWER 10 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1997:165266 HCAPLUS Full-text

DOCUMENT NUMBER:

126:157183

TITLE:

Process for the continuous preparation of 3-(methylthio)propanal from acrolein and

methyl mercaptan

INVENTOR(S):

Hsu, Yung C.

PATENT ASSIGNEE(S):

Novus International, Inc., USA

SOURCE:

PCT Int. Appl., 85 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent English

LANGUAGE:

FAMILY ACC. NUM. COUNT:

4

PATENT INFORMATION:

PA1	CENT	NO.			KIND DATE			APPLICATION NO.						DATE	
 WO	9700	- 858			A1		1997	0109	1	WO	1996-	US10	920		1996 0621
		DK, LK, PL, KE,	EE, LR, PT, LS,	ES, LS, RO, MW,	FI, LT, RU, SD,	GB, LU, SD, SZ,	GE, LV, SE, UG,	HU, MD, SG, AT,	IS, MG, SI BE,	BY JP MK CH	< , CA, , KE, , MN,	KG, MW, DK,	KP, MX, ES,	KR, NO, FI,	KZ, NZ, FR,
US	5905	CM,	GA,	GN,	ML						1996-			00,	1996
AU	9663	959			A1		1997	0122	i		< 1996-	6395	9		0620
	AU 726921 EP 842149										<	1996 0621			
	AU 726921 EP 842149				B2 20001123 A1 19980520					EP	1996-	9234	52		1996 0621
		BE,			ES,	FR,	GB,	IT,	LU,	NL	<	-			
CN	1188	470			Α		1998	0722	(1996-:	1949	43		1996 0621
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RU	2172	734			C2		2001	0827]		< 1998-:	1005	90		0621
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ES	ES 2192607			Т3		2003	1016	1		1996-! <	9∠ 34¦	o∠		1996 0621	
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CASREACT 126:157183 OTHER SOURCE(S):

In the title process, a liquid reaction, medium containing 3-(methylthio) propanal and a catalyst for the reaction between Me mercaptan and acrolein, is contacted with a gaseous acrolein feed stream in a gas-liquid contact zone. The gaseous acrolein feed stream comprises acrolein vapor and noncondensable gas and the acrolein is transferred from the acrolein feed stream to the reaction medium. Me mercaptan, introduced into the reaction medium, reacts with the acrolein in that medium, producing a liquid reaction product containing 3-(methylthio)propanal. The noncondensable gas is then separated from the liquid reaction product the reaction product is divided into a produce fraction and a circulating fraction, and the circulating fraction is recycled to the gas/liquid contact zone. Process flow diagrams are presented.

ΙT 3268-49-3P, 3-(Methylthio)propanal

> (process for the continuous preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IT 74-93-1, Methanethiol, reactions

> (process for the continuous preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

RN74-93-1 HCAPLUS

Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IC ICM C07C319-18 ICS C07C323-22

23-14 (Aliphatic Compounds)

Section cross-reference(s): 45, 48

TT **3268-49-3P**, 3-(Methylthio)propanal

> (process for the continuous preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

IT 74-93-1, Methanethiol, reactions 107-02-8, Acrolein, reactions

> (process for the continuous preparation of 3-(methylthio)propanal from acrolein and Me mercaptan)

L29 ANSWER 11 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER:

1997:111227 HCAPLUS Full-text

DOCUMENT NUMBER:

126:117741

TITLE:

Processes and catalysts for the preparation of

3-(methylthio)propanal and

2-hydroxy-4-(methylthio)butanenitrile

INVENTOR(S):

Blackburn, Thomas F.; Pellegrin, Paul F.;

Kranz, Allen H.

PATENT ASSIGNEE(S):

Novus International, Inc., USA

SOURCE:

PCT Int. Appl., 36 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

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	WO	9640	631			A1		1996	1219	Ţ	WO :	1996-	us90	60			1996 0604	
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OTHER SOURCE(S): MARPAT 126:117741

AB 3-(Methylthio)propanal (I) is prepared by the addition reaction of MeSH with acrolein, 2-hydroxy-4-(methylthio)butanenitrile is prepared by the addition reaction of I with HCN, and both reactions are conducted in the presence of an addition reaction catalysts comprising ≥1 organic base(s) (e.g., triisopropanolamine, nicotinamide, imidazole, benzimidazole, 2-fluoropyridine, poly-4-vinylpyridine, 4-dimethylaminopyridine, picoline, pyrazine, trialkylamines, etc.).

IT 64-19-7, Acetic acid, reactions 74-93-1,
Methanethiol, reactions 107-02-8, Acrolein, reactions
(processes and catalysts for the preparation of 3(methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

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RN 74-93-1 HCAPLUS CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

RN 107-02-8 HCAPLUS CN 2-Propenal (9CI) (CA INDEX NAME)

 $H_2C \longrightarrow CH \longrightarrow CH \longrightarrow O$

MeS-CH2-CH2-CHO

IC ICM C07C319-18 ICS C07C319-20; C07C323-22; C07C323-60

CC 23-19 (Aliphatic Compounds)
Section cross-reference(s): 45, 67

IT 64-19-7, Acetic acid, reactions 74-90-8, Hydrogen cyanide, reactions 74-93-1, Methanethiol, reactions 107-02-8, Acrolein, reactions 7664-38-2, Phosphoric acid, reactions 7664-93-9, Sulfuric acid, reactions (processes and catalysts for the preparation of 3- (methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile)

IT 3268-49-3P, 3-(Methylthio)propanal (processes and catalysts for the preparation of 3-(methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile)

L29 ANSWER 12 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1996:537082 HCAPLUS Full-text

DOCUMENT NUMBER:

125:167345

TITLE:

Preparation of 2-hydroxy-4-

(methylmercapto) butyric acid from acrolein and methyl mercaptan without using sulfuric acid

INVENTOR(S):

Matsuoka, Kazuyuki Daicel Chem, Japan

PATENT ASSIGNEE(S): SOURCE:

Jpn. Kokai Tokkyo Koho, 5 pp.

CODEN: JKXXAF

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
 JP 08157447	A2	19960618	JP 1993-159132	
			<	1993 0629
JP 3169103	. B2	20010521		
PRIORITY APPLN. INFO.:			JP 1993-159132	1993 0629

AB MeS(CH2)2CH(OH)CO2H (I), which is used as a feed additive, is prepared from CH2:CHCHO and MeSH, via MeS(CH2)2CHO, MeS(CH2)2CH(OH)CN (II), MeS(CH2)2CH(OH)CONH2 (III), and esters of MeS(CH2)2CH(OH)CO2H. Hydration of II in aqueous Me2CO in the presence of MnO2 at 60° for 6 h gave 89.0% III, which was autoclaved with MeOH and Pb nitrate at 170° and 20 kg/cm2 for 5 h with removing NH3 to afford MeS(CH2)2CH(OH)CO2Me at 83% conversion and 85% selectivity. Hydrolysis of the ester with Amberlyst 15 in H2O at 95° for 5 h gave I at 98.8% conversion and 97.1% selectivity.

IT 3268-49-3P, 3-(Methylmercapto)propionaldehyde

(preparation of hydroxy(methylmercapto)butyric acid from acrolein and Me mercaptan without using sulfuric acid)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

 ${\tt H3C-SH}$

IC ICM C07C323-52

ICS C07C319-18; C07C319-20

CC 23-17 (Aliphatic Compounds)

Section cross-reference(s): 17

IT 3268-49-3P, 3-(Methylmercapto)propionaldehyde

17773-41-0P, 2-Hydroxy-4-(methylthio)butyronitrile 49540-21-8P,

2-Hydroxy-4-(methylthio)butyramide 52703-96-5P

(preparation of hydroxy(methylmercapto)butyric acid from acrolein

and Me mercaptan without using sulfuric acid)

IT **74-93-1**, Methyl mercaptan, reactions 107-02-8,

2-Propenal, reactions

(preparation of hydroxy(methylmercapto)butyric acid from acrolein and Me mercaptan without using sulfuric acid)

L29 ANSWER 13 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1996:252233 HCAPLUS Full-text

DOCUMENT NUMBER:

124:288769

TITLE:

Preparation of 3-(methylthio)propanal

INVENTOR(S):

Hsu, Yung C.; Ruest, Dennis A. Novus International, Inc., USA

PATENT ASSIGNEE(S):

PCT Int. Appl., 70 pp.

SOURCE:

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.			KIND DATE					APPL	D <i>P</i>	ME					
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R: BE, DE, DK, ES, FR, GB, IE, IT, LU, MC, NL, PT

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AB The title process comprises condensation of CH2:CHCHO from a feed stream in a gas/liquid contact zone containing MeSCH2CH2CHO, MeSH, and catalyst, separation of non-condensable material from the feed stream, and withdrawal of liquid which is divided into a product stream and a stream which is returned to the gas/liquid contact zone.

IT 3268-49-3P, 3-(Methylthio)propanal

(preparation of 3-(methylthio)propanal).

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IC ICM C07C323-50 ICS C07C323-51

CC 23-14 (Aliphatic Compounds)

IT **3268-49-3P**, 3-(Methylthio) propanal

(preparation of 3-(methylthio)propanal)
IT 74-93-1, Methanethiol, reactions 107-02-8, Acrolein,
reactions

(preparation of 3-(methylthio)propanal)

L29 ANSWER 14 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 1995:472534 HCAPLUS Full-text

DOCUMENT NUMBER: 122:289231

TITLE: Thermal generation of flavor compounds from

thiamin and various amino acids

AUTHOR(S): Guentert, M.; Bertram, H. -J.; Hopp, R.;

Silberzahn, W.; Sommer, H.; Werkhoff, P.

CORPORATE SOURCE: Haarmann & Reimer GmbH, Corporate Research,

Holzminden, D-3450, Germany

SOURCE: Recent Dev. Flavor Fragrance Chem., Proc. Int.

Haarmann Reimer Symp., 3rd (1993),

215-40. Editor(s): Hopp, Rudolf; Mori, Kenji.

VCH: Weinheim, Germany.

CODEN: 60ZGAH

DOCUMENT TYPE: Conference LANGUAGE: English

Aqueous solns. of pure thiamin hydrochloride as well as mixts. of thiamin hydrochloride with cysteine hydrochloride and of thiamin hydrochloride with methionine were heated in an autoclave. The resulting flavor compds. were obtained by the simultaneous distillation/extraction procedure according to Likens and Nickerson. The concs. were presepd. by medium-pressure liquid chromatog. on silica gel using a pentane-diethyl ether gradient. The fractions were subsequently analyzed by capillary gas chromatog. (HRGC) and capillary gas chromatog.-mass spectrometry (HRGC/MS). Various unknown compds. were isolated by preparative capillary gas chromatog. in microgram quantities to elucidate their structures by IR and NMR spectroscopy and to check their olfactory properties. The spectroscopic data and sensory impressions are given. Most of the analyzed flavor compds. were synthesized. In particular, a series of new heterocyclic S-containing constituents with interesting taste properties was identified and confirmed by synthesis. Most of the degradation flavor compds. of pure thiamin contained sulfur and/or nitrogen. Many of these were heterocyclic constituents. The resp. mixts. with cysteine or methionine still led mainly to the formation of these thiamin degradation compds. but there was a distinctive addnl. effect caused by the main degradation products of the two amino acids which are hydrogen sulfide and mercaptoacetaldehyde (from cysteine) as well as methanethiol and methional (from methionine). The direct comparison between the flavor patterns of pure thiamin and of thiamin with cysteine and methionine resp., made it possible to study the influences of the different precursors on the resulting flavor compds. The explanation for the occurrence of the identified volatiles resulted in various proposals for the formation pathways of the thermal degradation reactions. To investigate the sensorially most relevant flavor compds. of the different mixts. aroma extract dilution analyses (AEDA) according to Grosch were made. The flavor dilution factors (FD) were calculated and are discussed. Addnl., the taste threshold values of various S-containing compds. in water were determined

IT 74-93-1, Methanethiol, biological studies

(formation and reaction of amino acid degradation products in food model)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

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(thermal generation of flavor compds. from thiamin and various
       amino acids)
RN
     64-19-7 HCAPLUS
CN
    Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)
RN
     3268-49-3 HCAPLUS
     Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)
MeS-CH2-CH2-CHO
CC
    17-2 (Food and Feed Chemistry)
IT
    74-93-1, Methanethiol, biological studies 7783-06-4,
    Hydrogen sulfide, biological studies
        (formation and reaction of amino acid degradation products in food
     64-19-7, Acetic acid, formation (nonpreparative)
ΙT
     137-00-8, Sulfurol 600-14-6, 2,3-Pentanedione
                                                      624-92-0,
    Dimethyl disulfide 656-53-1 2527-76-6, 2-Methyl-3-
     thiophenethiol 3268-49-3, Methional 3658-80-8,
     Dimethyltrisulfide 3760-25-6 4124-63-4, Mercaptoacetaldehyde
     5616-51-3, 2-Methyl-1,3-dithiolane 5756-24-1,
     Dimethyltetrasulfide 17042-24-9, 2-Mercapto-3-pentanone
     19788-49-9, Ethyl 2-mercaptopropanoate 26486-13-5,
     2-Methyl-4,5-dihydro-3-furanthiol 28588-74-1,
     2-Methyl-3-furanthiol 28588-75-2, Bis-(2-methyl-3-
                     31331-53-0, 1-(Methylthio)ethanethiol
     furyl)disulfide
     34047-39-7, 4-(Methylthio)-2-butanone 40789-98-8,
     3-Mercapto-2-butanone 65505-17-1, 2-Methyl-3-(methyldithio)furan
     67633-97-0, 3-Mercapto-2-pentanone 85196-66-3
                                                     91265-97-3
        (thermal generation of flavor compds. from thiamin and various
       amino acids)
L29 ANSWER 15 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN
                        1995:183935 HCAPLUS Full-text
ACCESSION NUMBER:
DOCUMENT NUMBER:
                        122:9491
TITLE:
                        Continuous process for preparation of
                        3-(methylthio)propanal from a gaseous acrolein
                        feed stream
                        Hsu, Yung C.; Ruest, Dennis A.
INVENTOR(S):
                        Novus International, Inc., USA
PATENT ASSIGNEE(S):
SOURCE:
                        U.S., 16 pp.
                        CODEN: USXXAM
DOCUMENT TYPE:
                        Patent
LANGUAGE:
                        English
FAMILY ACC. NUM. COUNT:
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PATENT NO. KIND DATE APPLICATION NO. DATE

PATENT INFORMATION:

US	5352837			Α		1994	1004		US	1993	-737	63							
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JP RU EP EP AT	R: AT, NL, 09501145 2118314 889029 889029 R: BE, 178594	PT,	SE	DE, T2 C1 A2 A3 ES, E	DK,	ES, 1997 1998 1999 2002 GB, 1999	FR, 0204 0827 0107 0313 IT, 0415	LU,	GR, JP : RU : EP : NL, AT :	, IE 1993 1998 , MC 1993 1993	-501 -100 -114 , PT -922	709 238 518 , :	9 8 8 1 1	LU,	MC, 199 090 199 090 199 090	93 09 93 09 93 09			

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US 1999-470407

1999 1222

AB A process for the continuous preparation of 3-(methylthio)propanal. A liquid reaction medium is contacted with a gaseous acrolein feed stream in a gas/liquid contact zone. The reaction medium contains 3-(methylthio)propanal. Me mercaptan and a catalyst for the reaction between Me mercaptan and acrolein. The gaseous acrolein feed stream comprises acrolein vapor and noncondensable gas. Acrolein is transferred from the acrolein feed stream to the reaction medium and reacts with Me mercaptan in that medium to produce a liquid reaction product containing 3-(methylthio)propanal. The non-condensable gas is separated from the liquid reaction product. The reaction product is divided into a product fraction and a circulating fraction, and the circulating fraction is recycled to the gas/liquid contact zone.

IT 3268-49-3P, 3-(Methylthio)propanal

> (continuous process for preparation of 3-(methylthio)propanal from a gaseous acrolein feed stream)

3268-49-3 HCAPLUS RN ·

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CH0

IT **74-93-1**, Methyl mercaptan, reactions (continuous process for preparation of 3-(methylthio)propanal from a gaseous acrolein feed stream)

74-93-1 HCAPLUS RN

Methanethiol (8CI, 9CI) (CA INDEX NAME) CN

H3C-SH

IC ICM C07C323-50 ICS C07C323-51

INCL 568041000

23-14 (Aliphatic Compounds)

Section cross-reference(s): 45

IT 3268-49-3P, 3-(Methylthio)propanal

(continuous process for preparation of 3-(methylthio)propanal from a gaseous acrolein feed stream)

IT 74-93-1, Methyl mercaptan, reactions

> (continuous process for preparation of 3-(methylthio)propanal from a gaseous acrolein feed stream)

L29 ANSWER 16 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 1994:161921 HCAPLUS Full-text

DOCUMENT NUMBER: 120:161921

TITLE: Microwave and thermally induced Maillard

reactions

AUTHOR(S): Yaylayan, V. A.; Forage, N. G.; Mandeville, S. CORPORATE SOURCE: Dep. Food Sci. Agric. Chem., McGill Univ.,

Ate. Anne de Bellevue, QC, H9X 3V9, Can.

SOURCE: ACS Symposium Series (1994),

543(Thermally Generated Flavors), 449-56

CODEN: ACSMC8; ISSN: 0097-6156

DOCUMENT TYPE: Journal LANGUAGE: English

The effect of amino acid type on the generation of Maillard aromas under microwave irradiation in an open system was evaluated by mixing different combinations of amino acids with the same reducing sugars and characterizing the aromas produced as caramel, meaty, nutty, fragrant, vegetable and baked. The amino acids were divided into five categories: aliphatic, aromatic, basic, acidic and sulfur-containing Certain trends emerged after anal. of the results that relate the presence of a specific amino acid category in the reaction mixture to a corresponding aroma note produced after microwave heating. The presence of amino acids with alkyl side chains was essential for the generation of caramel notes, sulfur-containing amino acids for meaty type notes and basic amino acids for nutty and baked notes. Selected formulations were also subjected to conventional heating and their sensory properties and chemical composition (by GC/MS anal.) were compared to those from microwave treated samples. No significant differences were observed between the two samples.

IT 64-19-7, Acetic acid, reactions 74-93-1,
Methanthiol, reactions 3268-49-3, 3-Methylthio propanal
(formation of, in Maillard reaction, heat and microwave treatment in relation to)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

HO-C-CH3

RN 74-93-1 HCAPLUS CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

RN 3268-49-3 HCAPLUS CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

CC 17-2 (Food and Feed Chemistry)

IT 64-18-6, Formic acid, reactions 64-19-7, Acetic acid, reactions 67-47-0, HMF 74-93-1, Methanthiol, reactions 75-07-0, Acetaldehyde, reactions 78-84-2, 2-Methylpropanal 96-17-3, 2-Methylbutanal 98-00-0, 2-Furan methanol 98-01-1,

2-Furancarboxaldehyde, reactions 108-31-6, 2,5-Furandione, reactions 110-00-9, Furan 116-09-6, 1-Hydroxy-2-propanone 122-78-1, Benzene acetaldehyde 123-72-8, Butanal 142-08-5, 2-(1H)-Pyridinone 290-37-9, Pyrazine 497-23-4, 2-(5H)-Furanone 534-22-5, 2-Methylfuran 590-86-3, 3-Methylbutanal 616-43-3, 3-Methylpyrrole 620-02-0, 5-Methyl-2-furancarboxaldehyde 624-92-0, Dimethyl disulfide 1073-96-7 1192-62-7, 1-(2-Furanyl)ethanone 3268-49-3, 3-Methylthio propanal 3658-77-3 3658-80-8, Dimethyl trisulfide 28564-83-2 153315-58-3

(formation of, in Maillard reaction, heat and microwave treatment in relation to)

L29 ANSWER 17 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 1994:133858 HCAPLUS Full-text

DOCUMENT NUMBER: 120:133858

TITLE: Process for producing 2-hydroxy-4-

methylthiobutanoic acid

INVENTOR(S): Matsuoka, Kazuyuki

PATENT ASSIGNEE(S): Daicel Chemical Industries, Ltd., Japan

SOURCE: PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
 WO 9323372	· A1	19931125	WO 1993-JP659	1993
W: US			<	0520
RW: BE, DE, FR,	GB			
JP 06049020	A2	19940222	JP 1993-143026	1993 0520
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JP 3219544 EP 601195	B2 A1		EP 1993-910360	
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EP 601195 R: BE, DE, FR,	B1 GB	19960828		
CN 1084511	Α	19940330	CN 1993-107598	1993 0521
			<	
CN 1036391 '	В	19971112	1004 170315	•
US 5386056	А	19950131	US 1994-178315	1994 0112
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PRIORITY APPLN. INFO.:			JP 1992-155802 A	1992 0521

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OTHER SOURCE(S): CASREACT 120:133858

A process for producing 2-hydroxy-4-methylthiobutanoic acid (I) together with methanol comprises hydrating 2-hydroxy-4- methylthiobutyronitrile (II) into 2hydroxy-4-methylthiobutanamide (III), reacting the amide with Me formate to yield Me 2-hydroxy-4-methylthiobutanoate (IV) and formamide, and hydrolyzing the Me ester. The discharge of a large amount of ammonium sulfate can be prevented, because no sulfuric acid is used as the reactant. The byproduct formamide and methanol are utilizable as the starting material of the reaction after converting them into HCN and Me formate, resp. Thus, addition of MeSH to acrolein in the presence of Cu(OAc)2 and hydroquinone and addition of the resulting 3-methylthiopropionaldehyde with HCN in the presence of NaOH in MeOH gave II. Hydration of II in the presence of MnO2 in aqueous acetone at 60° for 6 h to give III which was reacted with HCO2Me in MeOH containing MeONa to give IV and the byproduct formamide. Hydrolysis of IV in the presence of Amberlyst 15 in H2O at 95° gave I, while the byproduct MeOH was recovered. Formamide was fed into a stainless steel reactor packed with alumina at 500° to give HCN. MeOH was contacted with a catalyst prepared from Cu(NO3)2 and ammonium chromate in a stainless steel reactor to give Me formate.

IT 74-93-1, Methanethiol, reactions

(addition reaction of, with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P, 3-Methylthiopropionaldehyde

(preparation and addition of, with hydrogen cyanide)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

IC ICM C07C323-52

CC 23-16 (Aliphatic Compounds)

IT 74-93-1, Methanethiol, reactions

(addition reaction of, with acrolein)

IT 3268-49-3P, 3-Methylthiopropionaldehyde

(preparation and addition of, with hydrogen cyanide)

L29 ANSWER 18 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1992:135528 HCAPLUS Full-text

DOCUMENT NUMBER:

116:135528

TITLE:

Performance-oriented packaging standards;

changes to classification, hazard communication, packaging and handling

requirements based on UN standards and agency

initiative

CORPORATE SOURCE:

United States Dept. of Transportation,

SOURCE:

Washington, DC, 20590-0001, USA Federal Register (1990), 55(246),

52402-729, 21 Dec 1990

CODEN: FEREAC; ISSN: 0097-6326

DOCUMENT TYPE:

Journal

LANGUAGE: English

The hazardous materials regulations under the Federal Hazardous Materials Transportation Act are revised based on the United Nations recommendations on the transport of dangerous goods. The regulations cover the classification of materials, packaging requirements, and package marking, labeling, and shipping documentation, as well as transportation modes and handling, and incident reporting. Performance-oriented stds. are adopted for packaging for bulk and nonbulk transportation, and SI units of measurement generally replace US customary units. Hazardous material descriptions and proper shipping names are tabulated together with hazard class, identification nos., packing group, label required, special provisions, packaging authorizations, quantity limitations, and vessel stowage requirements.

IT 64-19-7, Acetic acid, miscellaneous 74-93-1, Methyl mercaptan, miscellaneous 107-02-8, 2-Propenal, miscellaneous 3268-49-3

(packaging and transport of, stds. for)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

74-93-1 HCAPLUS RN CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

RN 107-02-8 HCAPLUS CN 2-Propenal (9CI) (CA INDEX NAME)

H2C==CH-CH==O

RN 3268-49-3 HCAPLUS CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IT 50-00-0, Formaldehyde, miscellaneous 54-11-5, Nicotine 54-11-5D, Nicotine, compds. 55-63-0, Nitroglycerin 55-68-5, 56-18-8, 3,3'-Iminodipropylamine Phenylmercuric nitrate 56-23-5, miscellaneous 56-38-2, Parathion 57-06-7, Allyl isothiocyanate 57-14-7 57-24-9D, Strychnine, salts 60-00-4, EDTA, miscellaneous 60-24-2 60-29-7, Diethyl ether, miscellaneous 60-34-4, Methylhydrazine 60-57-1, Dieldrin 62-38-4, Phenylmercuric acetate 62-53-3, Aniline, miscellaneous 62-74-8, Sodium fluoroacetate 64-17-5, Ethanol, miscellaneous 64-18-6, Formic acid, miscellaneous 64-18-6D, Formic acid, chloro derivs. 64-19-7, Acetic acid, miscellaneous 64-67-5, Diethyl sulfate 66-25-1, Hexaldehyde 67-56-1, Methanol, miscellaneous 67-63-0, Isopropanol, miscellaneous 67-64-1, Acetone, miscellaneous 67-66-3, Chloroform, miscellaneous 68-11-1, Thioglycolic acid, miscellaneous 68-12-2, N,N-Dimethylformamide, miscellaneous 70-11-1, Phenacyl 70-30-4, Hexachlorophene 71-23-8, n-Propanol, miscellaneous 71-41-0, 1-Pentanol, miscellaneous 71-43-2, Benzene, miscellaneous 71-55-6, 1,1,1-Trichloroethane 74-83-9, miscellaneous 74-84-0, Ethane, Methane, miscellaneous 74-85-1, Ethylene, miscellaneous miscellaneous 74-86-2, Acetylene, miscellaneous 74-87-3, Methyl chloride, miscellaneous 74-88-4, Methyl iodide, miscellaneous 74-89-5, Methylamine, 74-90-8, Hydrogen cyanide, miscellaneous miscellaneous 74-93-1, Methyl mercaptan, miscellaneous 74-95-3, 74-96-4, Ethyl bromide Dibromomethane 74-97-5, Bromochloromethane 74-98-6, Propane, miscellaneous 75-00-3, 75-02-5, Vinyl fluoride Ethyl chloride 75-01-4, miscellaneous 75-04-7, Ethylamine, miscellaneous 75-05-8, Methyl cyanide, 75-07-0, Acetaldehyde, miscellaneous miscellaneous 75-09-2, Dichloromethane, miscellaneous Ethyl mercaptan 75-15-0, Carbon disulfide, miscellaneous 75-16-1, Methyl magnesium bromide 75-18-3, Dimethyl sulfide 75-19-4, 75-20-7, Calcium carbide Cyclopropane 75-21-8, Ethylene oxide, 75-21-8 75-25-2, Bromoform 75-26-3, miscellaneous 75-28-5, Isobutane 75-28-5D, Isobutane, mixts. 2-Bromopropane 75-29-6, 2-Chloropropane 75-31-0, Isopropylamine, miscellaneous 75-33-2, Isopropyl mercaptan 75-34-3, 1,1-Dichloroethane 75-35-4, miscellaneous 75-36-5, Acetyl chloride 75-38-7, 1,1-Difluoroethylene 75-39-8, Acetaldehyde ammonia Dichloromonofluoromethane 75-44-5, Phosgene 75-45-6, Chlorodifluoromethane 75-46-7, Trifluoromethane 75-50-3, 75-52-5, Nitromethane, Trimethylamine, miscellaneous 75-54-7, Methyldichlorosilane miscellaneous 75-55-8, 75-56-9, Propylene oxide, miscellaneous 75-59-2, Propylenimine Tetramethylammonium hydroxide 75-60-5, Cacodylic acid 75-61-6, Dibromodifluoromethane 75-63-8 75-71-8, Dichlorodifluoromethane 75-72-9, Chlorotrifluoromethane 75-73-0, Tetrafluoromethane 75-76-3, Tetramethylsilane 75-77-4, Trimethylchlorosilane, miscellaneous 75-78-5, Dimethyldichlorosilane 75-79-6, Methyltrichlorosilane 75-83-2 75-86-5, Acetone cyanohydrin 75-87-6, Chloral 75-91-2, tert-Butyl hydroperoxide 75-94-5, Vinyltrichlorosilane 76-01-7, Pentachloroethane 76-02-8, Trichloroacetyl chloride 76-05-1, Trifluoroacetic acid, miscellaneous 76-03-9, properties 76-06-2, Chloropicrin 76-06-2D, Chloropicrin, mixts. 76-15-3 76-16-4, Hexafluoroethane 76-19-7, Octafluoropropane 76-22-2, 77-47-4, Hexachlorocyclopentadiene 77-73-6 77-78-1, Dimethyl sulfate 78-00-2, Tetraethyl lead 78-10-4, Tetraethyl silicate 78-62-6, Dimethyldiethoxysilane 78-67-1,

Azodiisobutyronitrile 78-76-2, 2-Bromobutane 78-78-4, Isopentane 78-79-5, Isoprene, miscellaneous 78-81-9, Isobutylamine 78-82-0, Isobutyronitrile 78-83-1, Isobutanol, miscellaneous 78-84-2, Isobutyraldehyde 78-85-3, Methacrylaldehyde 78-87-5, Propylene dichloride 78-89-7, Propylene chlorohydrin 78-90-0, 1,2-Propylenediamine 2-Butanone, miscellaneous 78-94-4, Methyl vinyl ketone, miscellaneous 78-95-5, Monochloroacetone 79-01-6, Trichloroethylene, miscellaneous 79-03-8, Propionyl chloride 79-04-9, Chloroacetyl chloride 79-06-1, Acrylamide, miscellaneous 79-08-3, Bromoacetic acid 79-09-4, Propionic acid, miscellaneous 79-10-7, 2-Propenoic acid, miscellaneous 79-11-8, Chloroacetic acid, miscellaneous 79-20-9, Methyl 79-21-0, Peroxyacetic acid 79-22-1 acetate 79-24-3, 79-30-1, Isobutyryl 79-29-8, 2,3-Dimethylbutane Nitroethane chloride 79-31-2, Isobutyric acid 79-36-7, Dichloroacetyl chloride 79-38-9 79-41-4, miscellaneous 79-42-5 Dichloroacetic acid, miscellaneous 79-44-7, Dimethylcarbamoyl chloride 80-10-4, Diphenyldichlorosilane 80-15-9, Cumene hydroperoxide 80-17-1, Benzene sulfohydrazide p-Menthane hydroperoxide 80-51-3, Diphenyloxide-4,4'disulfohydrazide 80-56-8, α -Pinene 80-62-6 85-44-9, 1,3-Isobenzofurandione 82-71-3 86-50-0, Azinphos 87-90-1 methvl 87-68-3, Hexachlorobutadiene 88-17-5, 2-Trifluoromethylaniline 88-72-2, o-Nitrotoluene 88-73-3. o-Chloronitrobenzene 88-74-4, o-Nitroaniline 88-75-5, 88-89-1 89-58-7, p-Nitroxylene o-Nitrophenol 91-17-8, Decahydronaphthalene 91-20-3, Naphthalene, miscellaneous 91-20-3D, Naphthalene, diozonide derivs. 91-22-5, Quinoline, miscellaneous 91-59-8, β -Naphthylamine 91-66-7, N,N-Diethylaniline 92-52-4D, Biphenyl, chloro derivs. 92-52-4D, Biphenyl, halo derivs. 92-59-1, N-Ethyl-Nbenzylaniline 92-87-5, Benzidine 93-58-3, Methyl benzoate 94-17-7, p-Chlorobenzoyl peroxide 94-36-0, Benzoyl peroxide, miscellaneous 95-48-7, miscellaneous 95-50-1, o-Dichlorobenzene 95-54-5, o-Phenylenediamine, miscellaneous 95-55-6, o-Aminophenol 95-80-7 95-85-2, 2-Amino-4-chlorophenol 96-22-0, Diethyl ketone 96-12-8, Dibromochloropropane 96-23-1 96-24-2, Glycerol α -monochlorohydrin 96-32-2, Methyl bromoacetate 96-33-3 96-34-4, Methyl chloroacetate 96-37-7. Methyl cyclopentane 96-41-3, Cyclopentanol 97-62-1, Ethyl isobutyrate 97-63-2 97-64-3, Ethyl lactate 97-72-3, Isobutyric anhydride 97-85-8, Isobutyl isobutyrate 97-86-9 97-88-1 97-95-0 97-96-1, 2-Ethylbutyraldehyde 98-00-0, 98-01-1, Furfural, miscellaneous 98-07-7, Furfuryl alcohol Benzotrichloride 98-08-8, Benzotrifluoride 98-09-9, Benzene sulfonyl chloride 98-12-4, Cyclohexyltrichlorosilane 98-13-5, Phenyltrichlorosilane 98-16-8, 3-Trifluoromethylaniline 98-82-8, Isopropylbenzene 98-83-9, miscellaneous 98-85-1, α -Methylbenzyl alcohol 98-87-3, Benzylidene chloride 98-94-2 98-88-4, Benzoyl chloride 98-95-3, Nitrobenzene, 99-08-1, m-Nitrotoluene 99-09-2, m-Nitroaniline miscellaneous 99-35-4, Trinitrobenzene 99-99-0, p-Nitrotoluene 100-00-5 100-01-6, p-Nitroaniline, miscellaneous 100-02-7, p-Nitrophenol, miscellaneous 100-17-4 100-34-5, Benzene diazonium chloride 100-36-7, N, N-Diethylethylenediamine 100-37-8, Diethylaminoethanol 100-39-0, Benzyl bromide 100-41-4, Ethylbenzene, miscellaneous (packaging and transport of, stds. for)

IT 100-42-5, miscellaneous 100-44-7, Benzyl chloride, miscellaneous 100-47-0, Benzonitrile, miscellaneous 100-50-5, 1,2,3,6-Tetrahydrobenzaldehyde 100-57-2, Phenylmercuric hydroxide 100-61-8, N-Methylaniline, miscellaneous 100-63-0, Phenylhydrazine 100-66-3, Anisole, miscellaneous 100-73-2, 101-25-7, N,N'-Dinitrosopentamethylenetetramine Acrolein dimer 101-68-8 101-77-9, 4,4'-Diaminodiphenyl methane 101-83-7, Dicyclohexylamine 102-69-2, Tripropylamine 102-70-5, Triallylamine 102-81-8, Dibutylaminoethanol 102-82-9, Tributylamine 103-65-1, n-Propylbenzene 103-69-5, N-Ethylaniline 103-71-9, Phenylisocyanate, miscellaneous 103-80-0, Phenylacetyl chloride 103-83-3, Benzyldimethylamine 104-15-4, Toluene sulfonic acid, miscellaneous 104-51-8, 104-78-9 104-75-6, 2-Ethylhexylamine Butylbenzene 104-90-5, 2-Methyl-5-ethylpyridine 105-36-2 105-37-3, Ethyl propionate 105-39-5, Ethyl chloroacetate 105-48-6, Isopropyl chloroacetate 105-54-4, Ethyl butyrate 105-56-6, Ethyl cyanoacetate 105-57-7, Acetal 105-58-8, Diethyl carbonate 105-64-6, Isopropyl peroxydicarbonate 105-74-8, Lauroyl peroxide 106-31-0, Butyric anhydride 106-44-5, p-Cresol, miscellaneous 106-46-7, p-Dichlorobenzene 106-50-3, p-Phenylenediamine, miscellaneous 106-51-4, 2,5-Cyclohexadiene-1,4-dione, miscellaneous 106-63-8, Isobutyl acrylate 106-68-3, Ethyl amyl 106-88-7, 1,2-Butylene oxide 106-89-8, miscellaneous 106-92-3, Allyl glycidyl ether 106-93-4, Ethylene dibromide 106-95-6, Allyl bromide, miscellaneous 106-96-7, 3-Bromopropyne 106-97-8, Butane, miscellaneous 106-97-8D, Butane, mixts. 106-99-0, 1,3-Butadiene, miscellaneous 107-00-6, Ethylacetylene 107-02-8, 2-Propenal, miscellaneous 107-05-1, Allyl chloride 107-06-2, Ethylene dichloride, miscellaneous 107-07-3, Ethylene chlorohydrin, miscellaneous 107-10-8. Propylamine, miscellaneous 107-11-9, Allylamine 107-12-0, Propionitrile 107-13-1, Acrylonitrile, miscellaneous 107-14-2. Chloroacetonitrile 107-15-3, Ethylenediamine, miscellaneous 107-18-6, Allyl alcohol, miscellaneous 107-19-7, Propargyl 107-20-0, Chloroacetaldehyde 107-25-5, Vinylmethyl alcohol 107-29-9, Acetaldehyde oxime 107-30-2, Methylchloromethyl ether 107-31-3, Methyl formate 107-37-9, Allyltrichlorosilane 107-49-3, Tetraethyl pyrophosphate 107-71-1, tert-Butyl peroxylacetate 107-70-0 Amyltrichlorosilane 107-81-3, 2-Bromopentane 107-82-4, 1-Bromo-3-methylbutane 107-87-9, Methyl propyl ketone 107-89-1, Aldol 107-92-6, Butyric acid, miscellaneous 108-01-0, Dimethylethanolamine 108-05-4, Acetic acid ethenyl ester, miscellaneous 108-09-8, 1,3-Dimethylbutylamine 108-10-1, Methyl isobutyl ketone 108-11-2, Methyl isobutyl carbinol 108-18-9, Diisopropylamine 108-20-3, Diisopropyl 108-22-5, Isopropenyl 108-21-4, Isopropyl acetate acetate 108-23-6, Isopropyl chloroformate 108-24-7, Acetic anhydride 108-31-6, 2,5-Furandione, miscellaneous 108-39-4, 108-45-2, m-Phenylenediamine, miscellaneous miscellaneous 108-46-3, Resorcinol, miscellaneous 108-67-8, miscellaneous 108-77-0 108-83-8, Diisobutyl ketone 108-84-9 108-86-1, Benzene, bromo-, miscellaneous 108-87-2, Methyl cyclohexane 108-88-3, Toluene, miscellaneous 108-90-7, Chlorobenzene, miscellaneous 108-91-8, Cyclohexylamine, miscellaneous 108-94-1, Cyclohexanone, miscellaneous 108-95-2, Phenol, miscellaneous 108-98-5, Phenyl mercaptan, miscellaneous 109-02-4 109-09-1, 2-Chloropyridine 109-13-7, tert-Butyl peroxyisobutyrate 109-52-4, Valeric acid, miscellaneous

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109-53-5, Vinyl isobutyl ether 109-60-4, n-Propyl acetate
109-61-5, n-Propyl chloroformate 109-63-7, Boron trifluoride
diethyl etherate
                 109-65-9, n-Butyl bromide
                                              109-66-0, Pentane,
miscellaneous
               109-70-6, 1-Chloro-3-bromopropane
                                                   109-73-9,
n-Butylamine, miscellaneous 109-74-0, Butyronitrile 109-77-3,
              109-79-5, Butyl mercaptan 109-86-4, Ethylene
Malononitrile
glycol monomethyl ether 109-87-5, Methylal
                                              109-89-7,
Diethylamine, miscellaneous
                             109-90-0, Ethyl isocyanate
109-92-2, Vinyl ethyl ether
                             109-93-3, Divinyl ether
                                                      109-94-4,
                                        109-99-9,
Ethyl formate
               109-95-5, Ethyl nitrite
Tetrahydrofuran, miscellaneous
                               110-00-9, Furan
                                                 110-01-0,
                     110-02-1, Thiophene 110-12-3,
Tetrahydrothiophene
                     110-16-7, Maleic acid, miscellaneous
5-Methylhexan-2-one
110-18-9
          110-19-0
                     110-22-5, Diacetyl peroxide
                                                  110-43-0, Amyl
methyl ketone
               110-49-6
                         110-54-3, Hexane, miscellaneous
110-58-7, Amylamine
                     110-62-3, Valeraldehyde
                                              110-66-7, Amyl
mercaptan 110-68-9, N-Methylbutylamine
                                        110-69-0, Butyraldoxime
110-71-4, 1,2-Dimethoxyethane 110-74-7, Propyl formate
110-78-1, n-Propyl isocyanate 110-80-5, Ethylene glycol
monoethyl ether 110-82-7, Cyclohexane, miscellaneous 110-83-8,
Cyclohexene, miscellaneous
                           110-85-0, Piperazine, miscellaneous
110-86-1, Pyridine, miscellaneous
                                  110-87-2
                                             110-89-4,
Piperidine, miscellaneous 110-91-8, Morpholine, miscellaneous
110-96-3, Diisobutylamine 111-15-9, Ethylene glycol monoethyl
               111-34-2, Butylvinyl ether
ether acetate
                                            111-36-4, n-Butyl
            111-40-0
                      111-43-3, Dipropyl ether
isocyanate
                                                  111-49-9,
                   111-65-9, Octane, miscellaneous
Hexamethylenimine
                                                   111-69-3,
              111-71-7, n-Heptaldehyde
Adiponitrile
                                       111-76-2, Ethylene
glycol monobutyl ether 111-92-2, Di-n-butylamine 112-04-9
112-24-3, Triethylenetetramine
                               112-57-2 115-07-1, Propylene,
               115-10-6, Dimethyl ether
                                        115-11-7, Isobutylene,
miscellaneous
miscellaneous
               115-21-9, Ethyltrichlorosilane
                                                115-25-3,
Octafluorocyclobutane
                      116-14-3, Tetrafluoroethylene,
               116-15-4, Hexafluoropropylene 116-16-5,
miscellaneous
                   116-54-1, Methyl dichloroacetate 118-74-1,
Hexachloroacetone
                   118-96-7, Trinitrotoluene
Hexachlorobenzene
                                               120-92-3,
               121-43-7, Trimethyl borate
Cyclopentanone
                                           121-44-8,
Triethylamine, miscellaneous 121-45-9, Trimethyl phosphite
121-46-0, 2,5-Norbornadiene 121-69-7, N,N-Dimethylaniline,
miscellaneous
               121-73-3
                         121-82-4, Cyclotrimethylenetrinitramine
122-51-0, Ethyl orthoformate
                              122-52-1, Triethyl phosphite
                                   123-15-9
123-00-2, 4-Morpholinepropanamine
                                              123-19-3,
                123-20-6, Vinyl butyrate 123-23-9, Succinic.
Dipropylketone
acid peroxide
               123-30-8, p-Aminophenol
                                         123-31-9, Hydroquinone,
miscellaneous
               123-38-6, Propionaldehyde, miscellaneous
123-42-2, Diacetone alcohol
                            123-54-6, 2,4-Pentanedione,
miscellaneous
               123-62-6, Propionic anhydride
                                              123-63-7,
Paraldehyde
             123-72-8, Butyraldehyde 123-75-1, Pyrrolidine,
miscellaneous
               123-86-4, Butyl acetate 123-91-1, Dioxane,
               124-02-7, Diallylamine
miscellaneous
                                        124-09-4,
                                   124-13-0, Octyl aldehyde
Hexamethylenediamine, miscellaneous
124-18-5, n-Decane
                   124-38-9, Carbon dioxide, miscellaneous
124-40-3, Dimethylamine, miscellaneous
                                       124-41-4, Sodium
           124-43-6
                     124-47-0, Urea nitrate
                                               124-65-2, Sodium
           126-98-7, Methacrylonitrile
cacodylate
                                          126-99-8, Chloroprene
127-18-4, Tetrachloroethylene, miscellaneous
                                              127-85-5, Sodium
arsanilate
            129-79-3
                       131-52-2, Sodium pentachlorophenate
131-73-7, Hexanitrodiphenylamine
                                  131-74-8, Ammonium picrate
133-14-2
          133-55-1, N,N'-Dinitroso-N,N'-dimethyl terephthalamide
134-32-7, \alpha-Naphthylamine 138-86-3, Dipentene
                                                 138-89-6
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139-02-6, Sodium phenolate 140-29-4, Phenylacetonitrile 140-31-8, 1-Piperazineethanamine 140-80-7 140-88-5 141-32-2 (packaging and transport of, stds. for) 1303-33-9D, Arsenic sulfide, mixture with chlorates 1304-28-5, Barium oxide, miscellaneous 1304-29-6, Barium peroxide 1305-78-8, Calcium oxide, miscellaneous 1305-79-9, Calcium 1305-99-3, Calcium phosphide 1309-60-0, Lead dioxide peroxide 1310-58-3, Potassium hydroxide, miscellaneous 1310-65-2, Lithium hvdroxide 1310-73-2, Sodium hydroxide, miscellaneous 1310-82-3, Rubidium hydroxide 1312-73-8, Potassium sulfide 1313-60-6, Sodium peroxide 1313-82-2, Sodium sulfide, 1314-18-7, Strontium peroxide 1314-22-3, Zinc miscellaneous 1314-24-5, Phosphorus trioxide peroxide 1314-34-7, Vanadium 1314-56-3, Phosphorus pentoxide, miscellaneous trioxide 1314-62-1, Vanadium pentoxide, miscellaneous 1314-80-3, 1314-85-8, Phosphorus sulfide (P2S5) 1314-84-7, Zinc phosphide Phosphorus sesquisulfide 1319-77-3, Cresylic acid 1320-37-2, Dichlorotetrafluoroethane 1321-10-4, Chlorocresol 1321-31-9, 1327-53-3, Arsenic trioxide 1330-20-7, Xylene, Phenetidine 1330-45-6, Chlorotrifluoroethane miscellaneous 1330-78-5, 1331-22-2, Methyl cyclohexanone Tricresyl phosphate 1332-12-3, Fulminating gold 1332-37-2, Iron oxide, properties 1333-39-7, 1333-41-1, Picoline Phenolsulfonic acid 1333-74-0, Hydrogen, miscellaneous 1333-82-0, Chromium trioxide 1333-83-1, Sodium 1335-26-8, Magnesium peroxide hydrogen fluoride 1335-31-5, Mercury oxycyanide 1335-85-9, Dinitro-o-cresol 1336-21-6, 1337-81-1 1338-23-4, Methyl ethyl ketone Ammonium hydroxide 1341-24-8, Chloroacetophenone 1341-49-7, Ammonium hydrogen fluoride 1344-40-7, Lead phosphite, dibasic · 1344-67-8, Copper chloride 1498-40-4, Ethyl phosphonous 1498-51-7, Ethyl phosphorodichloridate dichloride 1569-69-3, Cyclohexyl mercaptan 1609-86-5, tert-Butyl isocyanate 1623-15-0 1623-24-1, Isopropyl acid phosphate 1634-04-4. Methyl-tert-butyl ether 1693-71-6, Triallyl borate 1705-60-8, 2,2-Di(4,4-di-tert-butylperoxycyclohexyl)propane 1712-64-7, Isopropyl nitrate 1719-53-5, Diethyldichlorosilane 1737-93-5, 3,5-Dichloro-2,4,6-trifluoropyridine 1789-58-8, Ethyldichlorosilane 1795-48-8, Isopropyl isocyanate 1838-59-1, Allyl formate 1873-29-6, Isobutyl isocyanate 1885-14-9, Phenylchloroformate 1947-27-9, Arsenic trichloride 2050-92-2, Di-n-amylamine 2094-98-6, 1,1'-Azodi (hexahydrobenzonitrile) 2144-45-8, Dibenzyl peroxydicarbonate 2155-71-7 2167-23-9, 2217-06-3, Dipicryl sulfide 2,2-Di(tert-butylperoxy)butane 2243-94-9, 1,3,5-Trinitronaphthalene 2244-21-5, Potassium 2294-47-5, p-Diazidobenzene 2312-76-7 dichloroisocyanurate 2338-12-7, 5-Nitrobenzotriazole 2487-90-3, Trimethoxysilane 2508-19-2, Trinitrobenzenesulfonic acid 2524-03-0, Dimethyl chlorothiophosphate 2524-04-1, Diethylthiophosphoryl chloride 2549-51-1, Vinyl chloroacetate 2551-62-4, Sulfur hexafluoride 2567-83-1, Tetraethylammonium perchlorate 2657-00-3, Sodium 2-diazo-1-naphthol-5-sulfonate 2691-41-0, Cyclotetramethylenetetranitramine 2696-92-6, Nitrosyl chloride 2699-79-8, Sulfuryl fluoride 2782-57-2, Dichloroisocyanuric acid 2782-57-2D, Dichloroisocyanuric acid, salts 2820-51-1, Nicotine hydrochloride 2825-15-2 2855-13-2, Isophoronediamine 2867-47-2, Dimethylaminoethyl methacrylate 2893-78-9, Sodium dichloroisocyanurate 2937-50-0, Allyl chloroformate 2980-64-5 Ethyl chlorothioformate 3025-88-5, 2,5-Dimethyl-2,5-dihydroperoxy hexane 3031-74-1, Ethyl hydroperoxide 3032-55-1 3054-95-3, 3,3-Diethoxypropene

3087-37-4, Tetrapropylorthotitanate 3129-90-6, Isothiocyanic 3129-91-7, Dicyclohexylammonium nitrite 3132-64-7, 3165-93-3, 4-Chloro-o-toluidine hydrochloride Epibromohydrin 3173-53-3, Cyclohexyl isocyanate 3179-56-4, Acetyl cyclohexanesulfonyl peroxide 3188-13-4, Chloromethyl ethyl ether 3248-28-0, Dipropionyl peroxide **3268-49-3** 3275-73-8, Nicotine tartrate 3282-30-2, Trimethylacetyl chloride 3497-00-5, Phenyl phosphorus thiodichloride 3689-24-5 3724-65-0, Crotonic acid 3811-04-9, Potassium chlorate 3926-62-3, Sodium chloroacetate 3982-91-0, Thiophosphoryl chloride 4016-11-9, 1,2-Epoxy-3-ethoxypropane 4098-71-9 4109-96-0, Dichlorosilane 4170-30-3, Crotonaldehyde 4300-97-4 4316-42-1, N-n-Butylimidazole 4419-11-8, 2,2'-Azodi(2,4dimethylvaleronitrile) 4421-50-5 4435-53-4, Butoxyl 4452-58-8, Sodium percarbonate 4472-06-4, Carbonazidodithioic 4484-72-4, Dodecyltrichlorosilane 4528-34-1 acid 4547-70-0 4682-03-5, Diazodinitrophenol 4795-29-3, 4591-46-2 Tetrahydrofurfurylamine 4904-61-4, 1,5,9-Cyclododecatriene 5283-66-9, Octyltrichlorosilane 5283-67-0, Nonyltrichlorosilane 5329-14-6, Sulfamic acid 5419-55-6, Triisopropyl borate 5610-59-3, Silver fulminate 5637-83-2, Cyanuric triazide 5653-21-4 5894-60-0, Hexadecyltrichlorosilane 5970-32-1, Mercury salicylate 6023-29-6 6275-02-1 6423-43-4 6427-21-0, Methoxymethyl isocyanate 6484-52-2, Nitric acid ammonium salt, properties 6484-52-2D, Ammonium nitrate, mixts. with fuel oils 6505-86-8, Nicotine sulfate 6659-60-5, 1,2,4-Butanetriol trinitrate 6842-15-5, Propylene tetramer 6867-30-7, Lithium acetylide ethylenediamine complex 7304-92-9 7332-16-3, Inositol hexanitrate 7429-90-5, Aluminum, miscellaneous 7429-90-5D, Aluminum, alkyl derivs. 7439-90-9, Krypton, miscellaneous 7439-92-1D, Lead, compds. 7439-93-2, Lithium, miscellaneous 7439-93-2D, Lithium, alkyl derivs. 7439-95-4, Magnesium, miscellaneous 7439-95-4D, Magnesium, alkyl 7439-97-6, Mercury, miscellaneous 7439-97-6D, Mercury, derivs. compds. 7440-01-9, Neon, miscellaneous 7440-09-7, Potassium, 7440-17-7, Rubidium, miscellaneous miscellaneous 7440-21-3, Silicon, miscellaneous 7440-23-5, Sodium, miscellaneous 7440-28-0D, Thallium, compds. 7440-29-1, Thorium, miscellaneous 7440-31-5D, Tin, organic compds. 7440-32-6, Titanium, properties 7440-36-0, Antimony, miscellaneous 7440-36-0D, Antimony, inorg. and organic compds. 7440-37-1, Argon, miscellaneous 7440-38-2, Arsenic, miscellaneous 7440-39-3, Barium, miscellaneous 7440-39-3D, Barium, alloys 7440-39-3D, Barium, compds. 7440-41-7, Beryllium, miscellaneous 7440-41-7D, Beryllium, 7440-43-9D, Cadmium, compds. 7440-44-0, Carbon, miscellaneous 7440-45-1, Cerium, miscellaneous 7440-46-2, Cesium, miscellaneous 7440-55-3, Gallium, miscellaneous 7440-58-6, Hafnium, miscellaneous 7440-59-7, Helium, 7440-61-1, Uranium, miscellaneous 7440-63-3, miscellaneous Xenon, miscellaneous 7440-66-6, Zinc, miscellaneous 7440-67-7, Zirconium, miscellaneous 7440-70-2, Calcium, miscellaneous 7440-70-2D, Calcium, alloys 7446-09-5, Sulfur dioxide, miscellaneous 7446-11-9, Sulfur trioxide, miscellaneous 7446-14-2, Lead sulfate 7446-18-6, Thallium sulfate Aluminum chloride (AlCl3), miscellaneous 7487-94-7, Mercuric chloride, miscellaneous 7488-56-4, Selenium disulfide 7521-80-4, Butyltrichlorosilane 7550-45-0, Titanium tetrachloride, miscellaneous 7570-26-5, 1,2-Dinitroethane 7572-29-4, Dichloroacetylene 7578-36-1 7580-67-8, Lithium hydride 7601-89-0, Sodium perchlorate 7601-90-3, Perchloric

acid, miscellaneous 7616-94-6, Perchloryl fluoride 7631-89-2, Sodium arsenate 7631-99-4, Sodium nitrate, miscellaneous 7632-00-0, Sodium nitrite 7632-51-1, Vanadium tetrachloride 7637-07-2, Boron trifluoride, miscellaneous 7645-25-2, Lead arsenate 7646-69-7, Sodium hydride 7646-78-8, Stannic chloride, miscellaneous 7646-85-7, Zinc chloride, miscellaneous 7646-93-7, Potassium hydrogen sulfate 7647-01-0, Hydrogen chloride, miscellaneous 7647-18-9, Antimony pentachloride 7647-19-0, Phosphorus pentafluoride 7664-38-2, Phosphoric acid, 7664-38-2D, Phosphoric acid, esters miscellaneous 7664-39-3, Hydrogen fluoride, miscellaneous 7664-41-7, Ammonia, miscellaneous 7664-93-9, Sulfuric acid, miscellaneous 7681-38-1, Sodium hydrogen sulfate 7681-49-4, Sodium fluoride, 7681-52-9, Sodium hypochlorite 7697-37-2, Nitric miscellaneous 7704-34-9, Sulfur, miscellaneous acid, miscellaneous (packaging and transport of, stds. for)

L29 ANSWER 19 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1990:630786 HCAPLUS Full-text

DOCUMENT NUMBER:

113:230786

TITLE:

Photochemical preparation of

3-(organothio)aldehydes from a mercaptan and

 α , β -unsaturated aliphatic aldehydes

INVENTOR(S):

Sandler, Stanley R. Pennwalt Corp., USA

SOURCE:

U.S., 3 pp. CODEN: USXXAM

DOCUMENT TYPE: '

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

TANIBI ACC. NOM. COONI.

PATENT INFORMATION:

PATENT ASSIGNEE(S):

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4944853	A	19900731	US 1989-405784	1989
				0911
			< 	
IN 173789	Α	19940716	IN 1990-CA292	
				1990
	٠.			0409
00101050			<	
JP 03184952	A2	19910812	JP 1990-94184	
				1990
				0411
EP 417386	7.1	10010220	< EP 1990-107565	
EP 41/300	AI	19910320	EP 1990-107565	1990
				0420
			<	0420
R: BE, CH, D	E. DK. E	S. FR. GR. T	T, LI, NL, SE	
AU 9053784				
3000,01			110 1330 33741	1990
				0423
			<	
AU 631202	B2	19921119		
BR 9001870	Α	19911112	BR 1990-1870	
				1990
				0423

US 1989-405784

1989 0911

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OTHER SOURCE(S): CASREACT 113:230786; MARPAT 113:230786

3-(Organothio)aldehydes R1CH(SR2)CH2CHO (I; R1 = H, C1-7 alkyl; R2 = C1-12 alkyl, C5-6 cycloalkyl, C6-12 aryl or alkaryl), useful as intermediates for the preparation of pesticides and antioxidants and as odorant or flavoring agents, are prepared by reaction of a mercaptan with substantially equimolar amount of α , β -unsatd. aliphatic aldehyde at .apprx.2°-60° in the absence of Ocontaining gas. Thus, a solution of 3.0 mol EtSH and 3.0 mol crotonaldehyde was cooled to 2-20° and was photolyzed in a 500 mL borosilicate reactor under the irradiation with a 450 W Hanovia high-pressure Hg lamp, while a slow stream of N was passed into the reactor. I (R1 = Me, R2 = Et) was obtained in 55.2% yield.

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P, 3-(Methylthio)propanal (preparation of, photochem. addition in)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

IC ICM B01J019-08

INCL 204157760

CC 23-14 (Aliphatic Compounds)

Section cross-reference(s): 5, 62

IT 74-93-1, Methyl mercaptan, reactions

(photochem. addition of, with acrolein)

IT **3268-49-3P**, 3-(Methylthio)propanal 27205-24-9P,

3-(Ethylthio)butanal

(preparation of, photochem. addition in)

L29 ANSWER 20 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1985:505316 HCAPLUS Full-text

DOCUMENT NUMBER:

103:105316

TITLE:

Methionine hydroxy analog or its derivative

and esters and 1-acyloxy-3-

hydrocarbylthiopropenes and products resulting

from their synthesis

INVENTOR(S):

Burrington, James David; Cesa, Mark Clark

PATENT ASSIGNEE(S):

Standard Oil Co., USA Eur. Pat. Appl., 30 pp.

SOURCE:

CODEN: EPXXDW

DOCUMENT TYPE:

LANGUAGE:

Patent English

FAMILY ACC. NUM. COUNT:

. 1

PATENT INFORMATION:

	PATENT NO.	KIND	DATE	API	PLICATION NO.		DATE
	EP 133796	A1	19850306	EP	1984-305228		1984
	EP 133796	D.1	10070204		<		0801
	R: BE, CH, DE,		19870304				
	US 4782173		19881101		1983-520042		
							1983
			•		<- -		0803
	CA 1251463	A1	19890321		1984-460202		
	011 1201100	711	13030321	O _I 1	1304 400202		1984
						•	0802
	TD C0100EE2	20	10050604		<		
	JP 60100553	AZ	19850604	JP	1984-163748		1984
							0803
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	BR 8403914	Α	19850709	BR	1984-3914		1984
							0803
					<		
	CA 1275111	A2	19901009	CA	1988-559288		1000
							1988 0218
					<		0210
PRIO	RITY APPLN. INFO.:			US	1983-520042	Α	
							1983 0803
					<		0803
					1984-460202	А3	
		•					1984
					· <		0802
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Methionine hydroxy analog or derivs. RSCH2CH2CH(OH)CO2H (I, R = C1-30 hydrocarbyl) were prepared by treating RSCH2CH2CHO with R1COX (R1 = H, C1-30 hydrocarbyl; X = R1CO2, F, Cl, Br, etc.), treating the resulting RSCH2CH:CHO2CR1 with CO and R2YH (R2 = H, C1-30 hydrocarbyl, Y = O; R2 = C1-30 hydrocarbyl, Y = S), and hydrolyzing the resulting RSCH2CH2CH(O2CR1)COYR2. Thus, MeSCH2CH2CHO was treated with Ac2O in the presence of KOAc at 145° for 3 h to give MeSCH2CH:CHOAc as a 43:57 Z/E mixture The latter mixture was treated with MeOH and CO in the presence of (Ph3P)2PdCl2 catalyst in a stainless steel bomb at 100° for 92.5 h to give 28.6% MeSCH2CH2CH(OAc)CO2Me (II) and 19.5% MeSCH2CH2CH(OAc)CSOMe. II was hydrolyzed by 2N HCl at 50° for 4 h to give 92% I (R = Me).

IT 64-19-7, uses and miscellaneous

(catalyst, for reaction of (methylthio)propionaldehyde with acetic anhydride)

RN 64-19-7 HCAPLUS

CN Acetic acid (7CI, 8CI, 9CI) (CA INDEX NAME)

ΙT 107-02-8P, preparation (formation of, from reaction of acetoxy(methylthio)propene with alcs. and carbon monoxide in absence of catalyst) 107-02-8 HCAPLUS RN 2-Propenal (9CI) (CA INDEX NAME) CN H2C-CH-CH-O IT 3268-49-3 (reaction of, with acetic anhydride) RN 3268-49-3 HCAPLUS CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME) $MeS-CH_2-CH_2-CHO$ IC ICM C07C149-20 CC 34-2 (Amino Acids, Peptides, and Proteins) Section cross-reference(s): 23 IT 64-19-7, uses and miscellaneous 110-86-1, uses and 121-44-8, uses and miscellaneous miscellaneous 563-67-7 603-35-0, uses and miscellaneous 1310-58-3, uses and miscellaneous 53189-26-7 (catalyst, for reaction of (methylthio)propionaldehyde with acetic anhydride) IT 79-20-9P **107-02-8P**, preparation (formation of, from reaction of acetoxy(methylthio)propene with alcs. and carbon monoxide in absence of catalyst) 3268-49-3 IT (reaction of, with acetic anhydride) L29 ANSWER 21 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 1985:487504 HCAPLUS Full-text DOCUMENT NUMBER: 103:87504 TITLE: Continuous preparation of β methylmercaptopropionaldehyde INVENTOR(S): Pavlovschi, Ana Maria; Levinta, Lucia; Gross, Gernot Holger PATENT ASSIGNEE(S): Combinatul Petrochimic, Pitesti, Rom. SOURCE: Rom., 2 pp. CODEN: RUXXA3 DOCUMENT TYPE: Patent LANGUAGE: Romanian FAMILY ACC. NUM. COUNT: PATENT INFORMATION:

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	RO 85095	В	19840924	RO 1982-106977	1982 0322
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PRIC	RITY APPLN. INFO.:			RO 1982-106977	1000
					1982 0322
				<	
AB	The addition react gave MeSCH2CH2CH0			th MeSH at atmospheric	pressure at 30-45°
IT	74-93-1 , reactions	g	yicias.		
DM	(addition of, wi	th acro	olein)		
RN CN	74-93-1 HCAPLUS Methanethiol (8CI,	9CI)	(CA INDEX NA	ME)	·
H3C	- SH				
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IT	3268-49-3P				
	(preparation of)				
RN CN	3268-49-3 HCAPLUS Propanal, 3-(methyl	thio)-	(9CT) (CA	TNDEY NAME!	
Civ	rropanar, 5 \meenyr	cnio,	(SCI) (CA	INDER NAME)	
				·	
MeS	-CH2-CH2-CHO				
IC	ICM C07C151-00				
CC	23-14 (Aliphatic Co	mpounds	s)		
IT	74-93-1, reactions	L1	2 - 4 - 3		
IT	(addition of, wi 3268-49-3P	th acro	olein)	•	
	(preparation of)				
ACCE	ANSWER 22 OF 35 HC SSION NUMBER: MENT NUMBER:		191700 HCAP	006 ACS on STN LUS <u>Full-text</u>	
TITL			:/ou : preparatio	n of B-	
			thiopropion		
	NTOR(S):			wachhofer, Ghislain	
SOUR	NT ASSIGNEE(S): CE:		Pat. Appl.,	ustries S. A., Fr. 13 pp.	
	· 		EPXXDW	FF'	
	MENT TYPE:	Patent			
	UAGE: LY ACC. NUM. COUNT:	French	1		
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KIND DATE

APPLICATION NO.

DATE

PATENT NO.

	EP	22697			A1	1981012	1	ΕP	1980-400951		
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	EP	22697			B1						
			CH,	DE,		GB, IT, NL			1070 17007		
	FR	2460925			A1	1981013	U	FК	1979-17827		1070
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		4319047			A	1982030		IIC	1980-164539		
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	BR	8004260			Α	1981012	7	BR	1980-4260		
		0001200			••	2302020	•		1000 1000		1980
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	ES	493224			A1	1981041	б	ES	1980-493224		
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	JP	56053648			A2	1981051	3	JP	1980-93336		1000
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	.TP	57008098			В4	1982021	5		_		
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AB Acrolein, prepared by air oxidation of propylene, was purified and treated with MeSH to yield MeSCH2CH2CHO in an apparatus which is described. The acrylic acid impurity was removed from the acrolein by countercurrent washing in water or solvent; the water was removed by condensation and the condensate was partially vaporized to recover acrolein.

IT **74-93-1**, reactions

(addition reaction of, with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

IC C07C149-14 23-14 (Aliphatic Compounds) CC **74-93-1**, reactions TT (addition reaction of, with acrolein) IT 3268-49-3P (preparation of) L29 ANSWER 23 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN 1978:109868 HCAPLUS Full-text ACCESSION NUMBER: DOCUMENT NUMBER: 88:109868 Illness with temporary work disability in TITLE: workers engaged in acrolein and methylmercaptopropionaldehyde (MMP) production Kantemirova, A. E. AUTHOR(S): CORPORATE SOURCE: USSR SOURCE: Trudy Volgogradskogo Gosudarstvennogo Meditsinskogo Instituta (1975), 26(4), 79-85 CODEN: TVLMB8; ISSN: 0376-141X DOCUMENT TYPE: Journal LANGUAGE: Russian AB Workers in the title industry are exposed to the action of the following air pollutants: methylmercaptan [74-93-1] 0.003-5.6, MMP [3268-49-3] 0.1-6.0, HCHO [50-00-0] 0.05-8.1, MeCHO [75-07-0] 0.48-22, and acrolein [107-02-8] 0.1-8.2 mg/m3. Catarrhal diseases are the most frequent and the highest sick rate is observed among women working for <1 or >7 y. 74-93-1, biological studies IT (health hazards of, in acrolein and methylmercaptopropionaldehyde manufacture) RN 74-93-1 HCAPLUS Methanethiol (8CI, 9CI) (CA INDEX NAME) CN ${\tt H3C-SH}$ IT 3268-49-3P (manufacture of, health hazards of) RN 3268-49-3 HCAPLUS CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME) MeS-CH2-CH2-CHO

CC 59-3 (Air Pollution and Industrial Hygiene)
 Section cross-reference(s): 23
IT 50-00-0, biological studies 74-93-1, biological studies
 75-07-0, biological studies

(health hazards of, in acrolein and methylmercaptopropionaldehyde manufacture)

107-02-8P, preparation **3268-49-3P** IT(manufacture of, health hazards of)

L29 ANSWER 24 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: DOCUMENT NUMBER:

86:120784

TITLE:

 $\beta ext{-Methylthiopropionaldehyde}$

INVENTOR(S):

Biola, Georges; Komorn, Yves; Limongi, Eric

PATENT ASSIGNEE(S):

Rhone-Poulenc S. A., Fr.

1977:120784 HCAPLUS Full-text

SOURCE:

Ger. Offen., 12 pp.

CODEN: GWXXBX

DOCUMENT TYPE:

Patent

LANGUAGE:

German

FAMILY ACC: NUM. COUNT:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
 DE 2627430	A1	19761223	DE 1976-2627430	
			52 25 0 252 133	1976
				0618
		•	<	
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DE 2627430	C3	19850110		
FR 2314917	A1	19770114	FR 1975-20183	. 1075
				1975 0620
•		•	<	0620
SU 691086	D	19791005	su 1976-2370202	•
50 031000	2	13,31003	50 1370 2070202	1976
				0615
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US 4225516	Α	19800930	US 1976-696432	
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JP 52003013	A2	19770111	JP 1976-70901	
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ES 448918	A1	19770701	ES 1976-448918	
DD 110310	•••	13,,0,01	ED 13/0 440310	1976
	•			0616
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BE 843077	A1	19761217	BE 1976-168033	
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NL 7606580	Α	19761222	NL 1976-6580	
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				0617
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NL 184517 NL 184517	· C	19890316 19890816		
SE 7607035	. С	19761221	SE, 1976-7035	
56 1001055	А	19/01221	36, 1970-7033	1976
				1370

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431089	В	19840116				
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7603949	Α	19770322	BR	1976-3949		
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						0618
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		•				0620
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AB The waste gas from acrolein (I) synthesis containing .apprx.5% I was freed from H2C:CHCO2H and H2O and dissolved in MeSCH2CH2CHO (II), then treated with MeSH at .apprx.30° to give MeSCH2CH2C(SMe)OH, which was maintained at .apprx.0.15% in the solution The combined yield of II was 99%.

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA:INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

IT **74-93-1**, reactions

(reaction of, with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IC C07C149-14

CC 23-14 (Aliphatic Compounds)

IT 3268-49-3P

(preparation of)

IT **74-93-1**, reactions

(reaction of, with acrolein)

L29 ANSWER 25 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1976:576769 HCAPLUS Full-text

DOCUMENT NUMBER:

85:176769

TITLE:

Development of a continuous method for

preparation of 3-(methylthio)propionaldehyde

AUTHOR(S):

Zvegintseva, G. B.; Medvedev, A. I.; Reimer,

M. I.; Dyadchenko, M. A.

CORPORATE SOURCE:

Nauchno-Issled. Inst. Khim. Polim. Mater.,

Tambov, USSR

SOURCE:

Tezisy Dokl. Nauchn. Sess. Khim. Tekhnol. Org.

Soedin. Sery Sernistykh Neftei, 13th (
1974), 343. Editor(s): Gal'pern, G.

D. "Zinatne": Riga, USSR.

CODEN: 33SUAA

DOCUMENT TYPE:

Conference

LANGUAGE:

Russian

AB A math. model was used to optimize a continuous process for MeSCH2CH2CHO (I) synthesis by reacting MeSH with acrolein (II); I was saturated with MeSH, and the resulting solution was treated with II in the presence of Et3N.

IT 74-93-1

(addition reaction of, with acrolein, (methylthio)propionaldehyde by, catalysis, simulation, and optimization of)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(by addition reaction of methanethiol with acrolein, catalysis, simulation, and optimization of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 ${\tt MeS-CH2-CH2-CHO}$

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction of, with acrolein, (methylthio)propionaldehyde by, catalysis, simulation, and optimization of)

IT 3268-49-3P

(by addition reaction of methanethiol with acrolein, catalysis, simulation, and optimization of)

L29 ANSWER 26 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1976:523278 HCAPLUS Full-text

DOCUMENT NUMBER:

85:123278

TITLE:

Peroxide initiation of the reaction of mercaptans with unsaturated compounds

AUTHOR(S):

Rykov, B. K.; Sizov, S. Yu.; Sukhanov, S. V.

CORPORATE SOURCE:

Volzh. Zavod. Org. Sint., Volzhsk, USSR

SOURCE:

Tezisy Dokl. Nauchn. Sess. Khim. Tekhnol. Org.

Soedin. Sery Sernistykh Neftei, 13th (1974), 343. Editor(s): Gal'pern, G.

D. "Zinatne": Riga, USSR.

CODEN: 33SUAA

DOCUMENT TYPE:

Conference

LANGUAGE:

Russian

AB RSH (R = lower alkyl, e.g., Me) addition to unsatd. compds. (e.g., acrolein) to give the corresponding sulfides (e.g., MeSCH2CH2CH0) was initiated by organic peroxides; α -haloacyl peroxides were recommended.

IT 74-93-1

(addition reaction with acrolein, initiator for)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

CC 23-9 (Aliphatic Compounds)

IT 74-93-1

(addition reaction with acrolein, initiator for)

IT 3268-49-3P

(preparation of)

L29 ANSWER 27 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:409198 HCAPLUS Full-text

DOCUMENT NUMBER:

83:9198

TITLE:

S-Substituted mercaptopropionaldehyde

INVENTOR(S):

Ito, Hiroo; Kimura, Kaoru; Yamada, Akira

PATENT ASSIGNEE(S):

Toa Gosei Chemical Industry Co., Ltd.

SOURCE:

Jpn. Tokkyo Koho, 3 pp.

CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
				-
JP 49024045	В4	19740620	JP 1970-43681	
				1970
				0523
			<	,
PRIORITY APPLN. INFO.:			JP 1970-43681	
INIONIII AIIIM. INIO			01 13/0 43001	1970
	•			0523
			<	

AB Cr(OAc)3.H2O and n-dodecylmercaptan were kept 1 hr at 30° with acrolein, containing a polymerization inhibitor (e.g. hydroquinone), to give 82.1% β -n-dodecylthiopropionaldehyde. The reaction of RSH (R = Me, Et, Bu, Ph) with

RCH:CR1CHO (R = H, Rl = H, Me; R = Me, Rl = H) and inorg. Cr salts were also discussed.

IT 74-93-1

(addition reaction with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IC C07C; B01J

CC 23-14 (Aliphatic Compounds)

IT **74-93-1** 75-08-1 108-98-5 109-79-5 112-55-0

(addition reaction with acrolein)

IT **3268-49-3P** 19378-51-9P 27098-65-3P 38160-52-0P

38160-57-5P 55154-14-8P

(preparation of)

L29 ANSWER 28 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:409197 HCAPLUS <u>Full-text</u> 83:9197

DOCUMENT NUMBER:

 β -Methylthiopropionaldehyde and its alkyl

derivatives

INVENTOR(S):

Ohuchi, Shunji; Shibuya, Kazumasa

PATENT ASSIGNEE(S):

Asahi Chemical Industry Co., Ltd.

SOURCE:

TITLE:

Jpn. Tokkyo Koho, 3 pp.

CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 49024046	B4	19740620	JP 1970-78498	
				1970
•				0909
			<	
PRIORITY APPLN. INFO.:	,	•	JP 1970-78498	
			•	1970
				0909
			<	

AB MeSH was added to RCH:CR1COR2 (R, R1, R2 = H, alkyl) in EtOH containing β -PhNHC10H7, NH4O2CNH2, NH4HCO3, (NH4)2CO3, NH4Cl-NaHCO3, or NH3-CO2 at 10-20° to give \leq 90% MeSCHRCHR1COR2.

IT 74-93-1

(addition reaction of, with acrolein, catalyst for)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(by addition reaction of methylmercaptan with acrolein, catalyst for)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IC C07C; B01J

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction of, with acrolein, catalyst for)

IT 3268-49-3P

(by addition reaction of methylmercaptan with acrolein, catalyst for)

L29 ANSWER 29 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:16324 HCAPLUS Full-text

DOCUMENT NUMBER:

82:16324

TITLE:

β-(Methylthio)propionaldehyde

INVENTOR(S):

Sizov, S. Yu.; Sukhanov, S. V.; Rykov, V. K.;

Shustov, V. I.; Tsarenko, S. V.

PATENT ASSIGNEE(S):

Volzhskii Plant of Organic Synthesis

SOURCE:

U.S.S.R. From: Otkrytiya, Izobret., Prom. Obraztsy, Tovarnye Znaki 1974, 51(34), 63.

CODEN: URXXAF

DOCUMENT TYPE:

Patent

LANGUAGE:

Russian

FAMILY ACC. NUM. COUNT:

1

PATENT NO.		KIND	DATE	APPLICATION NO.	DATE
· SU 443029		T	19740915	SU 1972-1819472	
					1972
					0810
				<	
PRIORITY APPLN.	INFO.:			SU 1972-1819472 A	
					1972
					0810

<--

AB MeSCH2CH2CHO (I) was prepared by treating acrolein with MeSH in an organic solvent (e.g., I) in 1:1 I-MeSH ratio.

IT 74-93-1

(addition reaction with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IC CO7C

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction with acrolein)

IT 3268-49-3P

(preparation of)

L29 ANSWER 30 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:16319 HCAPLUS <u>Full-text</u> 82:16319

DOCUMENT NUMBER: TITLE:

3-Methylmercaptopropionaldehyde

INVENTOR(S):

Koberstein, Edgar; Mueller, Klaus; Theissen,

Ferdinand

PATENT ASSIGNEE(S):

Deutsche Gold- und Silber-Scheideanstalt vorm.

Roessler

SOURCE:

Ger., 3 pp. CODEN: GWXXAW

DOCUMENT TYPE:

CODEN: GWA.

LANGUAGE:

Patent

LANGUAGE:

German

FAMILY ACC. NUM. COUNT:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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DE 2320544	B1	19740912	DE 1973-2320544	
			•	1973
•		•		0421
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DE 2320544	C2	19750605		
US 4048232	Α	19770913	US 1973-399127	
				1973
				0920
			<	
SU 505357	D	19760228	SU 1974-1996514	

				1974 0218	
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DD 110862	C	19750112	DD 1974-176862		
				1974	
			<	0228	
ES 423736	A1	19760416	ES 1974-423736		
		. =-		1974	
			•	0228	
GB 1400702		10750722	< CB 1074_0206		
	Α	19750723	GB 1974-9296	1974	
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·		•	<		
NL 7404691	Α	19741023	NL 1974-4691		
				1974	•
		•	<	0405	
BR 7402784	A0	19741105	BR 1974-2784	•	
				1974	
	•			0408	
СН 582665	А	19761215	< CH 1974-5019		
CH 302003	n	13/01213	CU 13/4-2013	1974	
				0410	
			<		
RO 68025	Р	19801230	RO 1974-78468	1074	
				1974 0418	
			<	0110	
BE 813990	A1	19741021	BE 1974-6044553		
				1974	
			<	0419	
FR 2226393	A1	19741115			
				1974	
				0419	
JP 50012012	A2	19750207	< JP 1974-44369 '		
OF JUUIZUIZ	<i>₽</i> ₩	13/3020/	OF 13/4-44303	1974	
				0419	
540000	_		<		
AT 7403268	Α	19751215	AT 1974-3268	1074	
				1974 0419	
			<- -	0413	
AT 331773	В	19760825			
IT 1005995	Α	19760930	IT 1974-50485	- 0	
				1974 0419 ·	
		•	<	0419	
CA 1005460	A1	19770215	CA 1974-197828		
				1974	
				0419	
SE 397344	D.	19771031	< SF 1074_5321		
3E 351344	В	19//1031	SE 1974-5321	1974	•
				0419	
			<		

Α

1973 0421

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AB CH2:CHCHO reacted with MeSH in the presence of hexamethylenetetramine catalyst to give 99.0-99.8% MeSCH2CH2CHO.

IT · 74-93-1

(addition reaction of, with acrolein, catalysts for)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IC CO7C

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction of, with acrolein, catalysts for)

IT 3268-49-3P

(preparation of)

L29 ANSWER 31 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:16318 HCAPLUS Full-text

DOCUMENT NUMBER:

82:16318

TITLE:

 β -Methylthiopropionaldehyde

INVENTOR(S):

Kojima, Takeshi; Horisawa, Toshiharu;

Shimasaki, Masami; Ito, Ryoichi

PATENT ASSIGNEE(S):

Kanegafuchi Chemical Industry Co., Ltd.

SOURCE:

Jpn. Tokkyo Koho, 2 pp. CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

1

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 49024890	В4	19740626	JP 1970-82267	
				1970
				0919
			<	
PRIORITY APPLN. INFO.:			JP 1970-82267	
		•		1970
				0919

AB Amino acids catalyzed the addition of MeSH (I) to CH2:CHCHO (II). Thus, 56 g II were added to 48 g I containing 0.5 g methionine at <40° over 60 min to give 93.6 g MeSCH2CH2CHO.

IT **74-93-1**

(addition reaction with acrolein, catalysts for)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

MeS-CH2-CH2-CHO

IC C07C; B01J

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction with acrolein, catalysts for)

IT 3268-49-3P

(preparation of)

L29 ANSWER 32 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1975:16317 HCAPLUS Full-text

DOCUMENT NUMBER:

82:16317

TITLE:

S-Substituted mercaptopropionaldehyde

INVENTOR(S):

Ito, Hiroo; Kimura, Kaoru; Sato, Masakatsu;

Yamada, Akira

PATENT ASSIGNEE(S):

Toa Gosei Chemical Industry Co., Ltd.

SOURCE:

Jpn. Tokkyo Koho, 3 pp. CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 49024454	В4	19740622	JP 1970-43680	
	•			1970
				0523
·			<	
PRIORITY APPLN. INFO.:			JP 1970-43680	
•				1970
		•		0523

AB The addition of RSH (R = alkyl) to R1CH:CR2CHO (R1, R2 = H, alkyl) to give RSCHR1CHR2CHO was promoted by strong acid catalysts, which activated the

double bond by protonating the CO group. Thus, CH2:CHCHO was added dropwise at 0-6.8° to MeSH and HCl, then held 1 hr at 30° to give 86.5% MeSCH2CH2CHO.

IT 74-93-1

(addition reaction with acrolein)

RN 74-93-1 HCAPLUS

CN Methanethiol (8CI, 9CI) (CA INDEX NAME)

H3C-SH

IT 3268-49-3P

(preparation of)

RN 3268-49-3 HCAPLUS

CN Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

 $MeS-CH_2-CH_2-CHO$

IC C07C; B01J

CC 23-14 (Aliphatic Compounds)

IT 74-93-1

(addition reaction with acrolein)

IT **3268-49-3P** 19378-51-9P

(preparation of)

L29 ANSWER 33 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

1973:57756 HCAPLUS Full-text

DOCUMENT NUMBER:

78:57756

TITLE:

Methional

INVENTOR(S):

Wakamatsu, Hachiro; Sato, Eiji; Sato,

Haruyoshi; Ono, Yoshio

PATENT ASSIGNEE(S):

Ajinomoto Co., Inc.

SOURCE:

Jpn. Tokkyo Koho, 5 pp.

CODEN: JAXXAD

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
			·.	
JP 47043925	В4	19721107	JP 1968-63178	
				1968 0903

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AB C6H6, MeOH, Me2CO, AcOEt, Et2O, tetrahydrofuran, Me2S, EtSMe, and n-heptane were used as solvents (0.4 l./mole MeSH) for the manufacture of MeSCH2CH2CHO (I) by treating MeSH, CH2:CH2, and CO in the presence of radical reaction initiators. Thus, 30 ml. n-heptane, 120 mg azobisisobutyronitrile, 0.64 g MeSH, and a gas mixture (665 kg/cm2) of CO and CH2:CH2 (25:1) was heated 2 hr at 80° to give 0.65 g I.

IT 3268-49-3P

(preparation of)

```
RN
     3268-49-3 HCAPLUS
CN
     Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)
 MeS-CH2-CH2-CHO
        (reaction of, with carbon monoxide and ethylene in solvents)
     74-93-1 HCAPLUS
RN
     Methanethiol (8CI, 9CI) (CA INDEX NAME)
 H3C-SH
IC
     C07C
CC
     23-14 (Aliphatic Compounds)
     3268-49-3P
        (preparation of)
IT
     74-93-1
        (reaction of, with carbon monoxide and ethylene in solvents)
L29 ANSWER 34 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                         1973:42803 HCAPLUS Full-text
DOCUMENT NUMBER:
                         78:42803
TITLE:
                         Formation of methional and methanethiol from
                         methionine
AUTHOR(S):
                         Wainwright, T.; McMahon, J. F.; McDowell, J.
CORPORATE SOURCE:
                         Res. Lab., Arthur Guinness Son and Co.
                         (Dublin) Ltd., Dublin, Ire.
SOURCE:
                         Journal of the Science of Food and Agriculture
                         (1972), 23(7), 911-14
                         CODEN: JSFAAE; ISSN: 0022-5142
DOCUMENT TYPE:
                         Journal
LANGUAGE:
                         English
AΒ
     Methanethiol is formed from methionine and sulfite in a reaction catalyzed by
     Fe or Mn(II) ions. Methionine sulfoxide and dimethyl disulfide are other
     products of the reaction. Methional [3-(methylthio)propional] is postulated .
     as an intermediate in the reaction.
     3268-49-3P
        (from decarboxylation of methionine)
RN
     3268-49-3 HCAPLUS
     Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)
CN
 MeS-CH2-CH2-CHO
```

IT

RN

CN

74-93-1P

74-93-1 HCAPLUS

(from decomposition of methionine)

Methanethiol (8CI, 9CI) (CA INDEX NAME)

CN

```
23-7 (Aliphatic Compounds)
CC
IT
     3268-49-3P
        (from decarboxylation of methionine)
TT
                624-92-0P
     74-93-1P
        (from decomposition of methionine)
L29 ANSWER 35 OF 35 HCAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER:
                         1967:463677 HCAPLUS Full-text
DOCUMENT NUMBER:
                         67:63677
TITLE:
                         Addition of methanethiol to some
                         \alpha, \beta-unsaturated aldehydes and study
                         of the reduction and acetalization of the
                         addition products
AUTHOR(S):
                         Boustany, Kamel S.
CORPORATE SOURCE:
                         Neuchatel Univ., Neuchatel, Switz.
SOURCE:
                         Journal of Chemistry of the U.A.R. (
                         1966), 9(3), 317-22
                         CODEN: JCURAS
DOCUMENT TYPE:
                         Journal
LANGUAGE:
                         English
     To 0.5 mole \alpha,\beta-unsatd. aldehyde at -20° is added a catalyst (piperidine or
     copper acetate) and 0.45 mole MeSH (previously chilled to -20^{\circ}), the mixture
     stirred 14 hrs. at 30-5°, extracted with Et20, washed with 2N HCl and H2O, the
     ethereal solution evaporated, and the residue distilled to give the
     corresponding 3-(methylthio)aldehyde (I). The I prepared were: 3-
      (methylthio)propanal, b14 61°, d20 1.036, n20D 1.4850, 2,4-
     dinitrophenylhydrazone, m. 121-2°; 3- methylthio)butanal, b10 63°, 0.997,
     1.4771 2,4-dinitrophenylhydrazone, m. 173-4°; and 3-(methylthio)-2-
     ethylhexanal, b0.2 74°, 0.954, 1.4759. Reduction of I with LiAlH4 in Et2O gave
     the corresponding alcs.: 3-(methylthio)propanol, b11 87°, 1.030, 1.4899; 3-
      (methylthio)butanol, b10 86°, 0.999, 1.4865; and 3-(methylthio)-2-
     ethylhexanol, b0.6 84°, 0.957, 1.4833. The alcs. treated with an excess of an
     acid or anhydride in the presence of p-toluenesulfonic acid gave the
     corresponding esters: 3-(methylthio)propyl acetate, bl4 96°, 1.041, 1.4636; 3-
      (methylthio)propyl butyrate, bll 108°, 0.994, 1.4580; 3-(methylthio)butyl
     formate, b9 78°, 1.039, 1.4680; and 3-(methylthio)-2-ethylhexyl formate, b0.2
     109°, 0.984, 1.4714. I treated with alc. (an excess of 50-100%) in the
     presence of p-toluenesulfonic acid, the reaction mixture refluxed 4-6 hrs.,
     extracted with Et2O, and worked up gave the corresponding 3-
      (methylthio) acetals: 3-(methylthio) propanal diethylacetal, b12 91°, 0.960,
     1.4525; 3-(methylthio)propanal glycol acetal, b11 93°, 1.107, 1.4839; 3-
      (methylthio)butanal, glycol acetal, b9 90°, 1.071, 1.4831; and 3-
      (methylthio) butanal dimethylacetal, b10 73°, 0.980, 1.4562. 3-(Methylthio)
     acids are prepared by addition of Me3SH to the corresponding unsatd. ester
     followed by saponification Thus, 3-(methylthio)butyric acid, b10 127°, 1.105,
     1.4833, was obtained from the corresponding ester. 2-(Methylthio) acetic
     acid, bl1 103°, 1.227, 1.4933, was prepared by reaction of MeSNa and ClCH2CO2H
     in the presence of aqueous NaOH.
IT
     3268-49-3P
        (preparation of)
RN
     3268-49-3 HCAPLUS
```

Propanal, 3-(methylthio)- (9CI) (CA INDEX NAME)

```
IT 74-93-1 (reaction of, with \alpha-unsatd. alkenals) RN 74-93-1 HCAPLUS CN Methanethiol (8CI, 9CI) (CA INDEX NAME)
```

H3C-SH

```
CC
    23 (Aliphatic Compounds)
IT
    505-10-2P 646-01-5P 2444-37-3P 3268-49-3P
                16630-52-7P 16630-53-8P
    7372-49-8P
                                             16630-54-9P
                                                           16630-55-0P
    16630-56-1P 16630-57-2P 16630-58-3P
                                              16630-59-4P
    16630-60-7P
                  16630-61-8P
                                16630-62-9P
                                              16630-64-1P
    16630-65-2P
                  16630-66-3P
                                18413-10-0P
        (preparation of)
IT
    74-93-1
       (reaction of, with \alpha-unsatd. alkenals)
```

=> fil casreact FILE 'CASREACT' ENTERED AT 09:12:47 ON 26 OCT 2006 USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT COPYRIGHT (C) 2006 AMERICAN CHEMICAL SOCIETY (ACS)

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FILE CONTENT:1840 - 22 Oct 2006 VOL 145 ISS 17

New CAS Information Use Policies, enter HELP USAGETERMS for details.

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations

database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance

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=> d que 110
L8 STR
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identification.

RRT PRO
H3C--SH OHC--C--S--CH3

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS

STEREO ATTRIBUTES: NONE

L10 12 SEA FILE=CASREACT SSS FUL L8 (15 REACTIONS)

=> d 110 1-12 ibib abs fhit

L10 ANSWER 1 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

145:166862 CASREACT Full-text

TITLE:

Catalytic method for the production of

3-(alkylthio)propanals from mercaptans and

glycerine

INVENTOR(S):

Redlingshoefer, Hubert; Weckbecker, Christoph;

Huthmacher, Klaus; Fischer, Achim; Barth,

Jan-Olaf

PATENT ASSIGNEE(S):

Degussa AG, Germany Ger. Offen., 7 pp.

SOURCE:

CODEN: GWXXBX

DOCUMENT TYPE:

Patent

LANGUAGE:

German

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	rent 1								A	PPLI	CATI	ON NO	0.	DATE	
									_						
DE	1020	0500	3990	A.	1	2006	0803		D	E 20	05-1	0200	5003	9902	0050128
WO	2006	0795	82	A.	1	2006	0803		W	20	06-E	P501	32	2006	0110
-	W:	ΑE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,
		CA,	CH,	CN,	co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	ΕE,	EG,
		ES,	FI,	GB,	GD,	GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,
						KP,									
		LY,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NA,	NG,	NI,	NO,	NZ,
						PT,						-			
		SY,	ТJ,	TM,	TN,	TR,	TT,	TZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,
			ZM,												•
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						LT,									
		SK,	TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,
		NE,	SN,	TD,	TG,	BW,	GH,	GM,	ΚE,	LS,	MW,	MZ,	NA,	SD,	SL,
		SZ,	TZ,	UG,	ZM,	ZW,	AM,	ΑZ,	BY,	KG,	KZ,	MD,	RU,	ТJ,	TM
US	2006														
US 2006183945 A1 20060817 US 2006-340673 20060127 PRIORITY APPLN. INFO.: DE 2005-10200500399020050128															

OTHER SOURCE(S):

MARPAT 145:166862

AB 3-(Alkylthio)propanals RSCH2CH2CHO [R = H, C1-3 alkyl; e.g., 3- (methylthio)propanal] are prepared in high yield and selectivity by the reaction of mercaptans RSH (e.g., Me mercaptan) with glycerol in the presence of catalysts (e.g., HZSM-5 zeolites).

RX(1) OF 1 A + B ===> C

RX(1) RCT A 56-81-5, B **74-93-1**

PRO C **3268-49-3** SOL 67-56-1 MeOH

CON 1 hour, 300 deg C, 61 bar

NTE reaction run in an autoclave, HZSM-5, Modul 28 zeolite catalyst used, catalyst calinated prior to use, reaction run at lower pressure for short time lead to side product formation, high pressure

L10 ANSWER 2 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

144:214741 CASREACT Full-text

TITLE:

Method and catalysts for preparing

3-(methylthio)propanal from acrolein and methyl mercaptan and for the manufacture of 2-hydroxy-4-(methylthio)butanenitrile from it

and hydrogen cyanide

INVENTOR(S):

Dubner, Frank; Weckbecker, Christoph

PATENT ASSIGNEE(S):

Germany

SOURCE:

U.S. Pat. Appl. Publ., 8 pp.

CODEN: USXXCO

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

1

PATENT	NO.		KI	ND	DATE			· A	PPLI	CATI	ON N	ο.	DATE	
				_ - -	:-			-						
US 200	60307	39	Α	1	2006	0209		U	S 20	05-1	9860	9	2005	0805
US 711	9233		В	2	2006	1010								
DE 102	00403	8053	Α	1	2006	0427		D	E 20	04-1	0200	4038	0532	0040805
WO 200	60156	84	A	2	2006	0216		W	0 20	05-E	P766	6	2005	0714
WO 200	60156	84	Α	3	2006	0803								
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	CA,	CH,	CN,	co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,
	ES,	FI,	GB,	GD,	GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,
	KE,	KG,	KM,	KP,	KR,	ΚZ,	LC,	LK,	LR,	LS,	LT,	LU,	LV,	MA,
	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NA,	NG,	NI,	NO,	NZ,	OM,	PG,

```
PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ,
             TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
        RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR,
            HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI,
             SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,
            NE, SN, TD, TG, BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL,
             SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
PRIORITY APPLN. INFO.:
                                          DE 2004-10200403805320040805
```

AB A method is described for preparing 3-(methylthio)propanal (I) by the the addition reaction of Me mercaptan to acrolein in the presence of macroreticular resin catalysts containing pendant tertiary-amine groups [e.g., [(dimethylamino)methyl]styrene copolymer] to give I which is then reacted with HCN in the presence of the same catalyst to give 2-hydroxy-4-(methylthio) butanenitrile. Process flow diagrams are presented.

RX(1) OF 6

RCT A 74-93-1 RX (1)

STAGE(1)

CAT 9040-03-3 Benzenemethanamine, ar-ethenyl-N,Ndimethyl-, homopolymer 10 minutes, 0 deg C

CON

STAGE(2)

RCT B 107-02-8

CON 2 hours, 0 deg C

14

PRO C **3268-49-3**

NTE solid-supported catalyst on Merrifield resin, 3-(methylthio)propanal used as reaction medium, batchwise synthesis

REFERENCE COUNT:

THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 3 OF 12 CASREACT COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 140:356948 CASREACT Full-text TITLE: Catalytic addition reaction for the production

of 3-(methylthio)propanal from mercaptomethane

and acrolein

Rey, Patrick INVENTOR(S):

Adisseo France S.A.S., Fr. PATENT ASSIGNEE(S): SOURCE: Eur. Pat. Appl., 10 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

LANGUAGE:

Patent English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

```
APPLICATION NO.
     PATENT NO.
                      KIND
                            DATE
                                                             DATE
                      A1
                            20040428
                                           EP 2002-356211
    EP 1413573
                                                             20021024
        R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE,
            MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ,
             EE, SK
    CA 2495746
                            20040506
                       AA
                                           CA 2003-2495746 20031014
    WO 2004037774
                       A1
                            20040506
                                           WO 2003-IB4557
                                                             20031014
            AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA,
             CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES,
             FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE,
             KG, KP, KR, KZ, LC, LK, LR; LS, LT, LU, LV, MA, MD, MG,
            MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO,
             RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ,
             UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
        RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
            AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ,
             DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL,
             PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN,
             GQ, GW, ML, MR, NE, SN, TD, TG
    AU 2003267771
                      A1 20040513
                                           AU 2003-267771
                                                             20031014
    EP 1556343
                           20050727
                      A1 .
                                           EP 2003-748466
                                                             20031014
           AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE,
            MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ,
             EE, HU, SK
    BR 2003015385
                            20050823
                                           BR 2003-15385
                       А
                                                             20031014
    CN 1705641
                                           CN 2003-80101589 20031014
                       Α
                            20051207
    JP 2006515834
                       T2
                            20060608
                                           JP 2004-546263
                                                             20031014
                                           US 2005-524548
    US 2005240048
                       A1
                            20051027
                                                             20050516
                                           NO 2005-2471
    NO 2005002471
                      Α
                            20050725
                                                             20050523
PRIORITY APPLN. INFO.:
                                           EP 2002-356211
                                                             20021024
                                           WO 2003-IB4557
                                                             20031014
```

AB A process for the production of 3-(methylthio)propanal comprises reacting mercaptomethane and acrolein in the presence of a catalyst comprising an organic base such as an N-alkylmorpholine (e.g., 4-methylmorpholine).

RX(1) RCT A 107-02-8, B **74-93-1**

PRO C 3268-49-3

CAT 64-19-7 AcOH, 109-02-4 N-Methylmorpholine

SOL 74-93-1 MeSH

CON SUBSTAGE(1) room temperature -> 40 deg C

SUBSTAGE(2) 40 deg C

NTE optimization study, optimized on catalyst

REFERENCE COUNT:

THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L10 ANSWER 4 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

140:287102 CASREACT Full-text

TITLE:

Method for producing 3-methylthiopropanal from

acrolein and methyl mercaptan

INVENTOR(S):

Shiozaki, Tetsuya; Haga, Toru

PATENT ASSIGNEE(S):

Sumitomo Chemical Company, Limited, Japan

SOURCE:

U.S. Pat. Appl. Publ., 4 pp.

CODEN: USXXCO

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

FAMILI ACC. NOM. COOMI.

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004063650	Al	20040401	US 2003-665006	20030922
JP 2004115461	A2	20040415	JP 2002-282874	20020927
EP 1408029	A1	20040414	EP 2003-21191	20030924
R: AT, BE,	CH, DE	, DK, ES, FR,	GB, GR, IT, LI, LU,	NL, SE,
MC, PT,	IE, SI	, LT, LV, FI,	RO, MK, CY, AL, TR	BG, CZ,
EE, HU,	SK			
CN 1496979	Α	20040519	CN 2003-125534	20030925
PRIORITY APPLN. INFO	.:		JP 2002-282874	20020927

AB 3-Methylthiopropanal is produced in high yield and selectivity by supplying acrolein and Me mercaptan together or sequentially with an acidic compound (e.g., acetic acid) and a basic compound (e.g., pyridine) into a reaction system to react the acrolein with the Me mercaptan, where the basic compound is used in an amount of about 0.3 mol or less per mol of the acidic compound

RX(1) RCT A 107-02-8, B **74-93-1**RGT D 64-19-7 AcOH
PRO C **3268-49-3**SOL 110-86-1 Pyridine
CON 45 - 50 minutes, 70 deg C
NTE other products detected

L10 ANSWER 5 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

128:114715 CASREACT <u>Full-text</u>

TITLE:

Processes for the preparation of

3-(methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile

INVENTOR(S):

Blackburn, Thomas F.; Pellegrin, Paul F.

PATENT ASSIGNEE(S): Novus International, Inc., USA

SOURCE:

U.S., 9 pp., Cont.-in-part of U.S. 5,663,409.

CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PA'	TENT	NO.		KI	ND	DATE								DATE	·
	5705													1995	1229
	5663														
	9604														
WO	9640	631		A	1	1996	1219		W	19	96-U	s906	0	1996	0604
	W:	AL,	AM,	AT,	ΑU,	AZ,	BB,	BG,	BR,	BY,	CA,	CH,	CN,	CZ,	DE,
		DK,	EE,	ES,	FI,	GB,	GE,	HU,	IS,	JP,	KE,	KG,	KP,	KR,	KZ,
		LK,	LR,	LS,	LT,	LU,	LV,	MD,	MG,	MK,	MN,	MW,	MX,	NO,	NZ,
		PL,	PT,	RO,	RU,	SD,	SE,	SG,	SI						
	RW:	KE,	LS,	MW,	SD,	SZ,	ŪG,	AT,	BE,	CH,	DE,	DK,	ES,	FI,	FR,
		GB,	GR,	ΙE,	IT,	LU,	MC,	NL,	PT,	SE,	BF,	ВJ,	CF,	CG,	CI,
		CM,	GA,	GN,	ML										
AU	9659	873		A.	1	1996	1230		ΑU	J 19	96-5	9873		1996	0604
	7141				_										
	8303								El	P 19	96-9	1722	2	1996	0604
EP	8303	41		B.	1	2001	0905								
						FR,									
	1189								, CI	1 19	96-1	9519	0	1996	0604
	1092														
	1151														
	2173														
	2160														
PT	8303	41		Т		2001	1228		P:	ր 19։	96-9	1722	2	1996	0604
	1510					2004	0707								
PRIORIT	Y APP	LN.	INFO	. :										1995	
														1995	
									W	199	96-U	S906	0	1996	0604

OTHER SOURCE(S): MARPAT 128:114715

AB A catalytic processes for the preparation of 3-(methylthio)propanal and 2-hydroxy-4-(methylthio)butanenitrile using novel addition catalysts is described. The novel addition catalysts include: triisopropanolamine, nicotinamide, imidazole, benzimidazole, 2-fluoropyridine, poly-4-vinylpyridine, 4-dimethylaminopyridine, picoline, pyrazine, trialkylamines, and tertiary amines. E.g., reaction of MeSH and acrolein in presence of poly-4-vinylpyridine gave 89.0% 3-(methylthio)propanal. The aldehyde product, containing the poly-4-vinylpyridine catalyst, was converted to the nitrile in the same reactor by treatment with HCN. The yield of nitrile was 72.9%.

RX(1) OF 3 A + B ===> C...

RX(1) RCT A 107-02-8, B **74-93-1**

PRO C 3268-49-3

CAT 110-86-1 Pyridine, 64-19-7 AcOH

NTE novel process focuses on the catalyst/acid combination;

process minimizes the extent of polymer formation

REFERENCE COUNT:

37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE

IN THE RE FORMAT

L10 ANSWER 6 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

126:157183 CASREACT Full-text

TITLE:

Process for the continuous preparation of 3-(methylthio)propanal from acrolein and

methyl mercaptan

INVENTOR(S):

Hsu, Yung C.

PATENT ASSIGNEE(S):

Novus International, Inc., USA

SOURCE:

PCT Int. Appl., 85 pp. CODEN: PIXXD2

DOCUMENT TYPE:

Patent

DOCUMENT TIPE

racent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATÉ
WO 9700858	A1	19970109	WO 1996-US10920	19960621

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W: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE,
             DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ,
             LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ,
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         RW: KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR,
             GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI,
             CM, GA, GN, ML
     US 5905171
                       Α
                             19990518
                                            US 1996-667099
                                                              19960620
                                            AU 1996-63959
    AU 9663959
                       Α1
                            19970122
                                                              19960621
    AU 726921
                       B2
                            20001123
     EP 842149
                       A1
                             19980520
                                            EP 1996-923452
                                                              19960621
     EP 842149
                       В1
                            20030205
             BE, DE, DK, ES, FR, GB, IT, LU, NL, MC, PT, IE
     CN 1188470
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                             19980722
                                            CN 1996-194943
                                                             19960621
     CN 1120834
                       В
                            20030910
     JP 11508266
                       T2
                            19990721
                                            JP 1997-504005
                                                              19960621
                       C2
     RU 2172734
                            20010827
                                            RU 1998-100590
                                                              19960621
     ES 2192607
                       Т3
                            20031016
                                            ES 1996-923452
                                                              19960621
PRIORITY APPLN. INFO.:
                                            US 1995-421P
                                                              19950622
                                            US 1996-667099
                                                              19960620
                                            WO 1996-US10920
                                                             19960621
```

AB In the title process, a liquid reaction, medium containing 3(methylthio)propanal and a catalyst for the reaction between Me mercaptan and acrolein, is contacted with a gaseous acrolein feed stream in a gas-liquid contact zone. The gaseous acrolein feed stream comprises acrolein vapor and noncondensable gas and the acrolein is transferred from the acrolein feed stream to the reaction medium. Me mercaptan, introduced into the reaction medium, reacts with the acrolein in that medium, producing a liquid reaction product containing 3-(methylthio)propanal. The noncondensable gas is then separated from the liquid reaction product the reaction product is divided into a produce fraction and a circulating fraction, and the circulating fraction is recycled to the gas/liquid contact zone. Process flow diagrams are presented.

$$RX(1) OF 1 A + B ===> C$$

RX(1) RCT A 107-02-8, B **74-93-1**PRO C **3268-49-3**NTE continous process

L10 ANSWER 7 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 124:184625 CASREACT <u>Full-text</u>
TITLE: Process for the treatment and condition

TITLE: Process for the treatment and conditioning of solid or liquid effluents charged with heavy

metals

INVENTOR(S):
Leybros, Jean

PATENT ASSIGNEE(S): Commissariat a l'Energie Atomique, Fr.

SOURCE:

Eur. Pat. Appl., 9 pp.

CODEN: EPXXDW

DOCUMENT TYPE:

Patent French

LANGUAGE:

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 687483	A1	19951220	EP 1995-401367	19950613
EP 687483	B1	19980826		
R: BE,	CH, DE, ES	, GB, IT, LI,	NL	
FR 2721237	A1	19951222	FR 1994-7297	19940615
FR 2721237	B1	19960802		
ES 2123221	Т3	19990101	ES 1995-401367	19950613
PRIORITY APPLN.	INFO.:		FR 1994-7297	19940615

AB The effluent is treated with a reducing agent (e.g., SO2) and then contacted with an organic extractant (e.g., bis(2- ethylhexyl)phosphoric acid) and a hydrocarbon (e.g., hydrogenated tetrapropylene) for selective removal of the metal ions, followed by removing the heavy metals from the organic extract by a 2nd aqueous extraction, and precipitating and filtering the metals from the aqueous solution

RX(1) OF 1

RX (1) RCT A 74-93-1, B 107-02-8

PRO C 3268-49-3

NTE Classification: S-Alkylation; "1,4-Addition"; # Conditions: (AcO)2; <50 deg 2atm; # Comments: 4.7.49

L10 ANSWER 8 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER:

120:133858 CASREACT Full-text

TITLE:

Process for producing 2-hydroxy-4-

methylthiobutanoic acid

INVENTOR(S):

Matsuoka, Kazuyuki

PATENT ASSIGNEE(S):

Daicel Chemical Industries, Ltd., Japan

SOURCE: PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
		'		
WO 9323372	A 1	19931125	WO 1993-JP659	19930520
W: US				

	RW	7: I	ΒE,	DE,	FR,	GB				•
· JI	060	490	020		A.	2	19940222	JP	1993-143026	19930520
JI	321	.954	44		B	2	20011015			
EI	601	.195	5		A.	1	19940615	EP	1993-910360	19930520
EI	601	.195	5		B	1	19960828		•	
	R:	I	ΒE,	DE,	FR,	GB				
Ch	108	45:	11		Α		19940330	CN	1993-107598	19930521
Ch	103	639	91		В		19971112			
US	538	605	56		, A		19950131	บร	1994-178315	19940112
PRIORIT	Y AF	PLI	1.	INFO.	. :			JP	1992-155802	19920521
		•						WO	1993-JP659	19930520

AB A process for producing 2-hydroxy-4-methylthiobutanoic acid (I) together with methanol comprises hydrating 2-hydroxy-4- methylthiobutyronitrile (II) into 2hydroxy-4-methylthiobutanamide (III), reacting the amide with Me formate to yield Me 2-hydroxy-4-methylthiobutanoate (IV) and formamide, and hydrolyzing the Me ester. The discharge of a large amount of ammonium sulfate can be prevented, because no sulfuric acid is used as the reactant. The byproduct formamide and methanol are utilizable as the starting material of the reaction after converting them into HCN and Me formate, resp. Thus, addition of MeSH to acrolein in the presence of Cu(OAc)2 and hydroquinone and addition of the resulting 3-methylthiopropionaldehyde with HCN in the presence of NaOH in MeOH gave II. Hydration of II in the presence of MnO2 in aqueous acetone at 60° for 6 h to give III which was reacted with HCO2Me in MeOH containing MeONa to give IV and the byproduct formamide. Hydrolysis of IV in the presence of Amberlyst 15 in H2O at 95° gave I, while the byproduct MeOH was recovered. Formamide was fed into a stainless steel reactor packed with alumina at 500° to give HCN. MeOH was contacted with a catalyst prepared from Cu(NO3)2 and ammonium chromate in a stainless steel reactor to give Me formate.

RX(2) OF 15 F + G ===> H...

RX(2) RCT F 107-02-8, G **74-93-1**PRO H **3268-49-3**CAT 123-31-9 Hydroquinone, 142-71-2 Cu(OAc)2
NTE 20°

L10 ANSWER 9 OF 12 CASREACT COPYRIGHT 2006 ACS on STN ACCESSION NUMBER: 114:207514 CASREACT Full-text

TITLE: Influence of phosphoric ester groups in

geranyldiphosphate biosynthesis

AUTHOR(S): Jacob, L.; Julia, M.; Pfeiffer, B.; Rolando,

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CORPORATE SOURCE:

Lab. Chim. 24, Ec. Norm. Super., Paris, 75231,

Fr.

SOURCE: Bulletin de la Societe Chimique de France

(1990), (Nov.-Dec.), 719-33 CODEN: BSCFAS; ISSN: 0037-8968

DOCUMENT TYPE: LANGUAGE: Journal English

AB A variety of elimination reactions of sulfonium salts related to intermediates in geranyl diphosphate biosynthesis confirmed that the direction of elimination is strongly influenced by the phosphate residue favoring the formation of the natural isomer. Thus, elimination of Me2S+CMe2CH2CH2OP(O)(OMe)2BF4- by NaOD-MeOD 2 h at 20° gave 72% natural isomer Me2C:CHCH2OP(O)(OMe)2 (I); elimination by Me3COK-DMSO-d6 gave 95% of an isomeric mixture containing 63% I and 37% CH2:CMeCH2CH2OP(O)(OMe)2. Elimination of Me2S+CRMeCH2CH2OP(O)(OMe)2 (R = prenyl) by NaOEt-EtOH gave 70% (glc) of a mixture containing 27% (E)-RCH2CMe:CHCH2OP(O)(OMe)2, 67% (Z)-RCH2CMe:CHCH2OP(O)(OMe)2, 4% RCH2C(:CH2)CH2CH2OP(O)(OMe)2, and 2% RCH:CMeCH2CH2OP(O)(OMe)2. Addnl. elimination reactions under solvolytic conditions were also carried out.

 $P(X(2)) ext{ OF } 39 ext{ D } + \textbf{E} ===> \textbf{F...}$

RX(2) RCT D 107-86-8, E **74-93-1** PRO F **133567-89-2**

L10 ANSWER 10 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 51:47157 CASREACT Full-text

TITLE: 3- (Methylthio) propanal

INVENTOR(S): Hunt, Madison; Merner, Richard R.

PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.

DOCUMENT TYPE: Patent
LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

AB A mixture of MeSH (I) 440 and pyridine 16 is fed into acrolein 500 and HOAc 5 parts in an autoclave below 75°. The final portion of 3-(methylthio)-propanal (II) and I is added rapidly at 40° to give 91-7% II.

RX(1) OF 1 A + B ===> C

RX(1) RCT A **74-93-1**, B 107-02-8

PRO C 3268-49-3

SOL 110-86-1 Pyridine, 64-19-7 AcOH

NTE Classification: S-Alkylation; "1,4-Addition"; # Conditions: MeSH pyridine AcOH; 70-75 deg; # Comments: high yield

L10 ANSWER 11 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 50:73727 CASREACT <u>Full-text</u>

TITLE: Sulfur-containing amino acids

AUTHOR(S): Reisner, David B.

CORPORATE SOURCE: Wallace & Tiernan, Inc., Newark, NJ

SOURCE: Journal of the American Chemical Society

(1956), 78, 2132-5

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

GI For diagram(s), see printed CA Issue.

AB MeCH: CHCHO (140 g.) and 96 g. MeSH in the presence of 2 drops of piperidine stirred 0.5 hr. at $5-10^{\circ}$ and 3 hrs. at room temperature, the mixture treated with an addnl. 28 g. MeSH, heated about 1 hr. at 90°, diluted with 500 cc. Et20, washed with dilute HCl and H20, dried, and evaporated, and the residue distilled gave 201 g. MeSCHMeCH2CHO (I), b23 80°. AcCH:CH2 (27 g.) and 18 g. MeSH yielded 35.4 g. Ac(CH2)2SMe, b55 106°, nD25 1.4711. I (48.5 g.), 113 g. (NH4)3SO3, 25.5 g. NaCN, 335 cc. EtOH, and 335 cc. H2O heated 5 hrs. with stirring at 55°, the mixture concentrated to about 300 cc., treated cautiously with 50 cc. concentrated HCl, heated 7 min. at about 90°, refrigerated, and filtered, and the residue washed with 200 cc. H2O yielded 49 g. 5-(β benzylmercapto) propylhydantoin, m. 117-18° (from EtOAc). Similarly were prepared the following compds. RR'C.CO.NH.CO.NH (R, R', m.p., and % yield given): MeS(CH2)2, Me, 109.5-10.5°, 93.8; MeSCHMeCH2, H, 191-2°, 50.1; MeSCHPhCH2, H, 173-4°, 491. S-Benzyl-4-methylhomocysteine (7.17 g.), m. $222.5-3.5^{\circ}$ (decomposition) (from H2O) (obtained in 94% yield from the hydantoin) (0.69,0.74, 0.93) (the figures given in parentheses through out this abstract represent the Rf values of the resp. compds. obtained by ascending paper chromatography with BuOH-AcOH, lutidine-collidine, and PhOH-H2O, resp.) in 300 cc. liquid NH3 treated with about 1.7 g. Na, the solution decolorized with about 1 g. NH4Cl, treated with 5 cc. MeI, and evaporated, the residue treated with 125 cc. H2O, washed with Et2O, filtered, neutralized with concentrated HCl to pH about 6, concentrated to about 50 cc., diluted with 50 cc. Me2CO, and refrigerated, and the crystalline deposit recrystd. from

aqueous MeOH yielded 4.1 g. MeSCHMeCH2CH(NH2)CO2H (II), m. 236-7° (decomposition), (0.44, 0.53, 0.79). Similarly were prepared: MeS(CH2)2CMe(NH2)CO2H, 61%, m. 284-5° (decomposition) (from aqueous MeOH), (0.45, 0.50, 0.77); MeSCHPh(CH2)2CH(NH2)CO2H, 49.3%, m. 201-2° (decomposition) (from H2O). BzCH2SMe (21.8 g.) in 50 cc. dry Et2O added with stirring to 1.4 g. LiAlH4 in 10 cc. dry Et20, the mixture refluxed 1 hr. with stirring, cooled, and treated with stirring with 200 cc. ice water and 100 cc. 5N H2SO4, the aqueous layer washed with Et2O, the combined Et2O solns. washed, dried, and evaporated under a jet of dry air, and the residue distilled gave 18.4 g. MeSCH2CH(OH)Ph (III), b1.8 113-14.5°. III (170 mg.) treated with MeI yielded III. MeI, m. 134-5° (decomposition). III (15.8 g.) in 25 cc. dry CHCl3 treated with cooling with 9.2 g. SOC12 in 15 cc. dry CHCl3, the mixture cooled 0.5 hr., kept at room temperature overnight and evaporated, the residue heated gently with 5 cc. dry CHCl3 and 5 cc. SOCl2, and the mixture distilled gave 14.3 g. MeSCH2CHClPh (IV), b2.8 106-7°, nD25 1.5692. AcNHCH(CO2Et)2 (11.6 g.) and 200 mg. KI added with stirring to 1.23 g. Na in 100 cc. absolute EtOH, the mixture treated with 10 g. IV in 1 portion, stirred 2 hrs. at room temperature, refluxed 5 hrs., and filtered hot, the residue washed with about 50 cc. hot EtOH, the combined alc. solns. evaporated to dryness in vacuo, the residual oil kept at room temperature overnight, and the crystalline material washed with dilute HCl and H2O and dried in vacuo over KOH pellets yielded 16 g. MeSCH2CHPhC(NHAc)(CO2Et)2 (V), m. 95-6° (from Et2O-pentane). Crude V (14.4 g.), 40 cc. H2O, and 10 cc. concentrated HCl refluxed 6 hrs. with stirring, the mixture treated with 40 cc. H2O and 10 cc. concentrated HCl, refluxed 1.5 hrs. with stirring, cooled to room temperature, the solid refluxed 8 hrs. with stirring with 80 cc. glacial AcOH and 10 cc. concentrated HCl, treated with Norit, and filtered, the residue washed with H2O, the combined filtrates evaporated in vacuo, the residue (about 10 g.) triturated with 50 cc. Me2CO and filtered, and the residue washed with Me2CO and dried yielded 5 g. MeSCH2CHPhCH(NH2)CO2H.HCl (VI.HCl), m. 208-9° (decomposition); the Me2CO solns. combined and evaporated to dryness, the residue refluxed 6.5 hrs. with 25 cc. H2O, 25 cc. glacial AcOH, and 10 cc. concentrated HCl, the solution evaporated to dryness in vacuo, the residue washed with Me2CO and neutralized with AmNH2, and a 1-g. portion dissolved in 8 cc. H2O and neutralized with AmNH2 to pH 6, diluted with 25 cc. Me2CO, and filtered, and the residue washed with 15 cc. Me2CO yielded 300 mg. VI; the filtrate diluted with Me2CO gave a 2nd crop, 350 mg. MeSH (14 g.) passed with stirring and cooling into 1.2 g. Na in 150 cc. absolute MeOH, the mixture treated with stirring and cooling with 50 q. Me α - benzamidosenecioate, diluted with 200 cc. absolute MeOH and 200 cc. dry C6H6, stirred 1 hr. at room temperature, allowed to stand overnight, treated with 3.12 g. glacial AcOH, and evaporated to dryness in vacuo at room temperature, the residue washed with warm dry C6H6, the C6H6 evaporated, the residue (58 g.), 300 cc. 85% HCO2H, 300 cc. concentrated HCl, and 300 cc. H2O refluxed 6 hrs., the solution concentrated to about 50 cc., washed with Et20, neutralized with AmNH2 to pH 6, diluted with 350 cc. Me2CO, and refrigerated 2 days, and the white crystals washed with 300 cc. Me2CO and 200 cc. Et2O yielded 16.8 g. S-methylpenicillamine, m. 281-2° (0.38, 0.50, 0.80); it was also obtained in the same manner from 2-phenyl-4-isopropylidene-5- oxazolone and 30 g. MeSH. MeSH (16 g.) passed into 1.2 g. Na in 300 cc. absolute MeOH, the solution treated with cooling and stirring with 62.3 g. 2phenyl-4-benzal-5-oxazolone in 500 cc. warm, dry C6H6, the mixture stirred about 1 hr., kept at room temperature, treated with 3.12 g. glacial AcOH, and evaporated to dryness in vacuo, the residue treated with 100 cc. warm C6H6 and filtered, the filtrate diluted with 100 cc. warm C6H6 and 500 cc. pentane, and chilled, and the deposit washed with 150 cc. pentane yielded 74 g. PhCH(SMe)CH(NHBz)CO2Me (VII), m. 97-8.5° (from EtOAc-pentane). Crude VII (32.9 g.) hydrolyzed with 150 cc. H2O, 150 cc. concentrated HCl, and 150 cc. 90% HCO2H, the solution concentrated in vacuo to near dryness, and the precipitate washed with three 100-cc. portions H2O, dissolved in 75 cc. H2O, neutralized to pH 6 with AmNH2, and chilled yielded 12.5 g. S-methyl-3phenylcysteine, m. $178-9^{\circ}$ (decomposition) (0.51, 0.65, 0.88). The following sulfoxides were prepared by oxidation of the appropriate sulfides with H2O2 by the method of Toennies and Kolb (C.A. 33, 5359.9) (% yield, m.p., and Rf values given): PhCH2S(0)CHMeCH2CH(NH2)CO2H, 64.7, 214-15° (decomposition) (from H2O), (0.45, 0.60, 0.92); MeS(O)CH2CH2CMe(NH2)CO2H, 91.8, 239.5-40.5° (decomposition) (from aqueous MeOH), (0.14, 0.35, 0.77); $\label{eq:Meson} \texttt{MeS}(\texttt{O})\,\texttt{CHMeCH2CH}\,(\texttt{NH2})\,\texttt{CO2H}\,\,\,(\texttt{VIII})\,,\,\,84.4\,,\,\,213.5\text{--}14.5\,^{\circ}\,\,\,(\texttt{from aqueous MeOH})\,,\,\,(0.13,0.13)$ 0.40, 0.80); MeS(O)CH2CHPhCH(NH2)CO2H, 74.4, 205-6° (decomposition) (from aqueous MeOH), (0.33, 0.59, 0.87); MeS(O)CHPhCH2CH(NH2)CO2H, 87.7, 189-90° (decomposition) (from aqueous MeOH), (0.33, 0.47, 0.85); Me2CHCH[S(0)Me]CH(NH2)CO2H, 77.7, 166-7° (from aqueous MeOH), (0.14, 0.40, 0.76); PhCH[S(O)Me]CH(NH2)CO2H, 73.2, $147-8^{\circ}$ (decomposition) (from aqueous MeOH), (0.29, 0.54, 0.82). VIII (600 mg.), 3 cc. H2O, 2 cc. MeOH, 0.2 cc. concentrated HCl, and 2 cc. 30% H2O2 refluxed 2 hrs., treated with 1 cc. 30% H2O2, refluxed again 2 hrs., neutralized with AmNH2 to pH 6.5, diluted with 100 cc. Me2CO and filtered, and the residue washed with 50 cc. Me2CO yielded 550 mg. MeS(O2)CHMeCH2CH(NH2)CO2H, m. 230-1° (decomposition) (from aqueous MeOH), (0.14, 0.50, 0.72). In the same manner was prepared PhCH2S(O2)CH2CH2CH(NH2)CO2H, 70.6%, m. 229-30° (decomposition) (from H2O), (0.50, 0.65, 0.84). The following sulfones were prepared by the oxidation on the appropriate sulfides with H2O2 in the presence of NH4 molybdate and HClO4 by the method of Toennies and Kolb (C.A. 35, 6571.1) (% yield, m.p., and Rf values given): MeS(O2)CH2CH2CMe(NH2)CO2H, 73.6, 288-9° (decomposition) (from aqueous MeOH), (0.16, 0.45, 0.65); MeS(O2)CH2CHPhCH(NH2).CO2H (IX), 50.8, 222-3° (decomposition) (from H2O), (0.32, 0.61, 0.79); MeS(O2)CHPhCH2CH(NH2)CO2H (X), 95.4, 196.5-7.5° (decomposition), (0.37, 0.55, 0.79); Me2CHCH[S(O2)Me]CH(NH2)CO2H, 77.7, 166-7° (from aqueous MeOH), (0.14, 0.53, 0.68); MeS(O2)CHPhCH(NH2)CO2H, 51.2, 141-2°(decomposition) (from aqueous MeOH), (0.30, 0.52, 0.70). VIII (6.0 g.) treated dropwise with stirring at 3° with 10.4 cc. concentrated H2SO4, the mixture heated with stirring to 45°, treated during 1 hr. at 48° with 54 cc. 1.4N HN3 in CHCl3, then heated with stirring 5 hrs. at 48°, treated with 13.5 cc. HN3 solution, heated 5 hrs. with stirring at 50°, stirred overnight at room temperature, poured with stirring onto 75 g. crushed ice, neutralized with solid Ba(OH)2 to about pH 2.5 then to pH 5 with solid BaCO3, and centrifuged, the supernatant decanted, the residue mixed with H2O, centrifuged, and decanted, this operation repeated until free of amino acid, the combined aqueous solns. concentrated in vacuo at 50° to about 100 cc., treated with C, and filtered, and the filtrate concentrated to about 40 cc., filtered, and evaporated to dryness yielded 6.4 g. MeS(:NH)CHMeCH2CH(NH2)CO2H, m. 199-200° (decomposition) (from aqueous MeOH), (0.08, 0.38, 0.71). In the same manner was prepared: MeS(:NH)CH2CH2CHMe(NH2)CO2H,100, 199-200° (decomposition) (from aqueous MeOH), (0.10, 0.35, 0.67). IX (100 mg.) treated with about 60 mg. N-bromosuccinimide gave MeS(O2)CH2CHPhCHO, isolated as the 2,4-dinitrophenylhydrazone, m. 188-9°. (decomposition). X gave similarly MeS(O2)CHPhCH2CHO, isolated as the 2,4dinitrophenylhydrazone, decomposed at $196-8^{\circ}$ with a change from yellow to red at 169°. Only 4 of the amino acids suppressed the multiplication of T2 bacteriophage of Escherichia coli strain A.T.C.C. number 11303 at pH 7 and 37° at 100 p.p.m. or less.

RX(2) RCT F 123-73-9, G 74-93-1

PRO H 16630-52-7

NTE Classification: S-Alkylation; "1,4-Addition"; #

Conditions: MeSH (piperidine); 5-10 deg 30mn; 3h 20 deg;

1h 90 deg

L10 ANSWER 12 OF 12 CASREACT COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 42:25284 CASREACT Full-text

TITLE: Synthesis of DL-methionine

AUTHOR(S): Pierson, Earl; Giella, Mario; Tishler, Max

CORPORATE SOURCE: Merck & Co., Inc., Rahway, NJ

SOURCE: Journal of the American Chemical Society

(1948), 70, 1450-1

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

AB Addition of 48 g. MeSH to 56 g. CH2:CHCHO and 0.5 g. Cu(OAc)2 at 35-40° gives 84% MeSCH2CH2CHO (I), bl1 52-4°, n20D 1.4850, d20 1.036 (2,4-dinitrophenylhydrazone, m. 116-19°). I (10.4 g.), shaken with 10.4 g. NaHSO3 in 35 mL. H2O, the product treated (in 3 portions) with 4.9 g. NaCN in 15 mL. H2O (temperature below 35°), the oil immediately extracted with C6H6, and the C6H6 extracted with NaHSO3, gives 90% α -hydroxy- β -

(methylmercapto) butyronitrile (II), an oil that distilled at $100^\circ/3~\mu$. I (26 g.), 113 g. (NH4)2CO3, 24.5 g. NaCN, 335 mL. EtOH, and 335 mL. H2O, heated 4 h. at $50-5^\circ$, and the filtrate concentrated to 300 mL. and heated 5 min. at 90° with 50 mL. concentrated HCl, give 79% 5-(2- methylmercaptoethyl) hydantoin (III), m. $103-5^\circ$; it results in 50% yield (based on I) from II and (NH4)2CO3 in 50% MeOH (2.5 h. at $50-5^\circ$). III (17.4 g.) and 8.8 g. NaOH in 75 mL. H2O, refluxed 6 h., an addnl. 4.4 g. NaOH added, and the refluxing continued for 18 h., give 10.6 g. DL-methionine (IV), m. 269° (decomposition); if I and III are not isolated, the yield (based on CH2:CHCHO) is 50%. II (123 g.), treated 30 min. at 60° with NH3, gives 40% of crude methionine nitrile, which could not be purified; hydrolysis by heating 5.5 h. on the steam bath with 20 mL. concentrated HCl yields 75% IV. Hydrolysis of III to IV was also effected by concentrated HCl at 135° and by (NH4)2S at 135° .

RX(1) RCT A 107-02-8, B **74-93-1**

RGT D 142-71-2 Cu (OAc) 2

PRO C **3268-49-3**

NTE Classification: "1,4-Addition"; S-Alkylation; # Conditions: Cu(OAc)2 MeSH gas; 30mm 40 deg; 1h